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Inorganic Analyses in Water Quality Control Programs: Instructor's Guide

(U.S.) National Training and Operational
Technology Center, Cincinnati, OH

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Instructor's Guide

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This manual is designed for use by instructors who will have to teach others how to perform analyses for selected inorganic pollutants. It is to be used in conjunction with the training manual bearing the same title (EPA-430/1-81-015). It includes topics related to the presentation of training courses in which the analyses would be taught. These topics include: the course description, personnel needed to conduct the training, the course agenda, a milestones list for planning and preparing the course, instructional resources available to support the instruction and a list of equipment and supply requirements. Also included are Instructional Package Worksheets (lesson plans) giving details relative to presenting instruction regarding each of the analyses covered in the associated student training manual.

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PREFACE

Introduction

This is one of several courses offered by the National Training and Operational Technology Center (NTOTC) which have been prepared in packaged form for use by the States in their training programs, and by other organizations having a need to present this type of training. Each course package consists of:

1. An instructor's guide containing material related to course planning and conduct,
2. a training manual, for use by the participants, containing the course subject matter and
3. supportive visual and audiovisual training aids used by NTOTC in presenting the course. The training aids are available on loan to those offering the course. The content of each slide is reproduced in this manual.

This guide reflects the manner in which the course has been offered by NTOTC. It is intended to assist the organization offering the training, and should not be considered as an inflexible method of presenting the course. Some may want to follow the format exactly as presented--others may not. In either case, this guide should prove helpful in reducing the amount of original developmental work required, and in suggesting methods and approaches when modifications of the course plan presented herein are being considered.

Organization of the Guide

This instructor's guide consists of two major parts. Part I contains information required for course planning and management. Part II consists of a series of Instructional Package Worksheets (IPW's) which set forth learning objectives and the instructional approach used by NTOTC for each topic included in the training manual.

Analytical Methodology for Regulatory Programs

It is essential that analytical procedures taught in the course conform to those prescribed for use in USEPA regulatory programs. These procedures are identified in regulations which appear first in the Federal Register, and which are later codified in Title 40 of the annual edition of the Code of Federal Regulations.

For the National Pollutant Discharge Elimination System (NPDES), Part 136 of Title 40, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", specifies methods to be used for the measurement of contaminants in wastewater.

For drinking water, Part 141 of Title 40, "National Interim Primary Drinking Water Regulations" (NIPDWR), specifies methods to be used in determining the level of contaminants in finished water.

The Instructional Package Worksheet on Compliance Methodology in Part II of this guide provides details concerning EPA regulations for these programs.

For additional information concerning this course, or other packaged courses, contact:

Director, NTOTC
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

PART I COURSE PLANNING AND MANAGEMENT

This section of the manual is concerned with the administrative aspects of planning, preparing, and conducting the course.

A. COURSE DESCRIPTION

This description was prepared for course presentation at the NTOTC and may be useful to others in course-related publicity.

INORGANIC ANALYSES IN WATER QUALITY CONTROL PROGRAMS

5 Days

This course is for chemists and technicians with little or no experience in inorganic analyses commonly required for the National Pollutant Discharge Elimination System (NPDES), the National Interim Primary Drinking Water Regulations (NIPDWR) and for other water quality programs. Applicants should have one year of college level inorganic chemistry and one semester of college level quantitative analysis (or equivalent). They should have basic laboratory skills including use of analytical balances, volumetric glassware and titration assemblies. They should be actively engaged in a water quality control program.

After successfully completing the course, the student will know the general classes of methods listed as approved in the Federal Register for analysis of inorganic pollutants and will be able to use the methods to measure each parameter selected for the course. He will also know how to apply quality assurance techniques to his work and how to validate his analytical accuracy and precision.

The training is a five-day course which includes classroom instruction, student performance of laboratory procedures, and discussion of each laboratory assignment and reported results.

The student will perform the test procedures for acidity and alkalinity (titrations to pH end points), hardness (EDTA titration), chlorine (amperometric back titration), total phosphorus (persulfate digestion, ascorbic acid reduction, colorimetry), fluoride (SPADNS colorimetry and ion electrode), nitrate and nitrite (cadmium reduction, colorimetry), total and suspended solids (gravimetric), turbidity (nephelometer), and specific conductance (Wheatstone bridge conductivity meter). Other topics are sample handling; compliance methodology; accuracy, precision and error of data; elements of quality assurance programs.

B. PERSONNEL

Some of the sections in Part I of this manual refer to various personnel associated with this training course when it is presented at the National Training and Operational Technology Center (NTOTC). Their course-related activities are listed below.

1. Training Supervisor -

   Has overall responsibility for the NTOTC training program.

2. Course Coordinator -

   Is responsible for all elements involved in planning and conducting a specific course.

3. Course Secretary -

   Performs all course-related clerical duties.

4. Instructor -

   For assigned topics, is responsible for planning instructional approach, developing instructional materials, and delivering the instruction. During laboratory sessions, another instructor may be designated to assist the (primary) instructor so that participants can be provided as much individualized attention as possible.

5. Laboratory Assistant -

   Assists in the preparation of laboratory reagents, assembles equipment and is available as required during laboratory exercises.

C. SUMMARY PLAN FOR THE COURSE AND COURSE SCHEDULE

A convenient format to use in the early stages of devising a course plan is a day-to-day assignment of time blocks based on estimates by instructors of the training time required for each parameter. (An example is on the next page.) Using available time as a first criterion will allow a variety of possible sequences. One such sequence, which has been successfully used by NTOTC to conduct this course in the past, begins on page 2-3. Examples of other considerations are:

1. If some equipment must be used in more than one test, schedule another topic between the two tests to allow time for the required clean-up.
2. Schedule the topics so each instructor alternates between prime and assistant responsibilities to allow time for preparations which must be done right before training sessions.
3. If one procedure requires skills taught in another procedure, order the presentations accordingly.

Although NTOTC presents all of these topics in a five day course, there is nothing to prevent your choosing individual topics for instruction. The Instructional Package Worksheet for each topic (in Part II of this Guide) represents an independent module. If any other topic is directly related, this will be stated in the "Entry Level Behavior" section (V) of the IPW.

Likewise, you can develop any time schedule convenient to your staff and students, e.g., weekly presentations of individual topics. The time block approach is very useful for planning such a series, and also the IPW section (VIII B) on "Sequencing" which includes a time breakdown for classroom and laboratory activities. The time allotments for each subject are very feasible, providing ample time for instruction in essentials. Thorough preparation by the instructor is imperative to make each instructional minute count.

SUMMARY PLAN FOR THE COURSE:
APPROXIMATE AGENDA FOR COURSE 100.4

MONDAY		TUESDAY		WEDNESDAY		THURSDAY		FRIDAY	
Activity	Time Hours	Activity	Time Hours	Activity	Time Hours	Activity	Time Hours	Activity	Time Hours
Opening, Objectives, Evaluation	1	Acidity and Alkalinity	1/3	Accuracy	2/3	Nitrate and Nitrite	3	Turbidity (cont.)	3/4
Sample Handling	1/2	Precision	1	Fluoride (continued)	1			Solids (cont.)	1/2
Compliance Meth.	1	Hardness and Chlorine (continued)	2 1/3	Phosphorus and Fluoride (continued)	1 1/2			Specific Conduct. (continued)	3/4
Laboratory Safety	1/2					Solids (continued)	1/2	Turbidity/Solids Specific Conduct. (continued)	1 3/4
Analytical Tech.	1/2								
Lunch	3/4	Lunch	3/4	Lunch	3/4	Lunch	3/4	Lunch	3/4
Volumetric Anal.	1	Hardness and Chlorine	1	Phosphorus and Fluoride	1 3/4	Solids (continued)	2 1/2	Turbidity Solids Specific Conduct.	1/2
pH	3/4	Phosphorus (continued)	1 1/2	Nitrate and Nitrite (continued)	2	Quality Assurance	1 1/4	Course Post-Evaluation and Closing	1
Acidity and Alkalinity (continued)	1 3/4	Spectrophotometer	3/4						
TOTAL	7 3/4	TOTAL	7 2/3	TOTAL	7 2/3	TOTAL	8	TOTAL	6

COURSE SCHEDULE:

INORGANIC ANALYSES IN WATER QUALITY CONTROL PROGRAMS (100.4)

(Location)
(Date)

AGENDA

Course Coordinator: ____________ Laboratory Assistant: ____________

DAY & TIME	SUBJECT	OUTLINE	INSTRUCTOR*
MONDAY			
8:15 - 8:30	Registration		Course Secretary
8:30 - 8:35	Welcome		Course Coordinator
8:35 - 8:50	Course Objectives Introductions		Course Coordinator
8:50 - 9:20	Pre-Course Evaluation		Course Coordinator
	I DATA COLLECTION & EVALUATION		
9:25 - 9:55	Sample Handling-Field through Laboratory	1	Instructor #1
9:55 -10:15	Break		
10:15 -11:15	Compliance Methodology	2	Instructor #2
11:20 -11:55	Laboratory Safety Practices	30	Instructor #3
	II VOLUMETRIC ANALYSES		
11:55 -12:15	Analytical Techniques		Instructor #1
12:15 - 1:00	Lunch		
1:00 - 1:55	Volumetric Analysis	6	Instructor #2
2:00 - 2:45	pH	7	Instructor #1
2:50 - 3:20	Acidity and Alkalinity	7, 8	Instructor #2
3:30 - 4:45	Laboratory		
	Acidity	17	Instructor #1
	Alkalinity	18	Instructor #2
TUESDAY			
8:30 - 9:00	Class Discussion: Methodology, Acidity and Alkalinity Data		Instructor #2
9:00 -10:00	Indeterminate Error-Precision	3,4,5	Instructor #2
10:00 -10:15	Break		
10:15 -10:55	Calcium & Magnesium Hardness	9	Instructor #3
11:00 -11:45	Chlorine Determinations	10	Instructor #2
11:45 -12:30	Laboratory		
	Group A - Hardness	19	Instructor #3
	Group B - Chlorine	20	Instructor #2

DAY & TIME	SUBJECT	OUTLINE	INSTRUCTOR*
12:30 - 1:15	Lunch		
1:15 - 2:00	Laboratory		
	Group A - Chlorine	20	Instructor #2
	Group B - Hardness	19	Instructor #3
2:00 - 2:15	Class Discussion: Hardness and Chlorine Data		Instructor #3 Instructor #2
2:15 - 2:30	Break		
	III PHOTOMETRIC ANALYSES-COLORIMETRY		
2:30 - 3:15	Phosphorus	11	Instructor #3
3:15 - 4:00	Laboratory		
	Digestion of Phosphorus Sample and Standards	22	Instructor #3 (Instructor #2)
4:00 - 4:45	Use of a Spectrophotometer Calibration Graphs	21	Instructor #3
WEDNESDAY			
8:30 - 9:10	Determinate Error-Accuracy	4, 5	Instructor #2
9:15 -10:15	Fluoride	12	Instructor #1
10:15 -11:50	Laboratory		
	Group A - Phosphorus pH Adjustment, Colorimetry	22	Instructor #3
	Group B - Fluoride	23, 24	Instructor #1
11:50 -12:35	Lunch		
12:35 - 2:10	Laboratory		
	Group A - Fluoride	23, 24	Instructor #1
	Group B - Phosphorus pH Adjustment, Colorimetry	22	Instructor #3
2:10 - 2:25	Class Discussion: Fluoride and Total Phosphorus Data		Instructor #1 Instructor #3
2:35 - 3:05	Nitrate and Nitrite Nitrogen	13	Instructor #2
3:05 - 3:15	Cadmium Reduction Columns	25	Instructor #2
3:15 - 4:45	Laboratory		
	Prepare Reduction Columns	25	Instructor #2 (Instructor #1)
THURSDAY			
8:30 - 9:00	Cadmium Reduction Procedure	25	Instructor #2
9:00 -10:45	Laboratory		
	Nitrate + Nitrite Nitrogen by Cadmium Reduction Method	25	Instructor #2 (Instructor #1)
10:45 -11:30	Class Calculations, Graphing Discussion: Nitrate & Nitrite Data		Instructor #2

DAY & TITLE	SUBJECT	OUTLINE	INSTRUCTOR*
THURSDAY (Cont'd.)	IV GRAVIMETRIC ANALYSES		
11:30 - 11:45	Solids	26, 27	Instructor #2
11:45 - 1:00	Group A - Weighings - Lunch	26, 27	Instructor #1
	Group B - Lunch - Weighings	26, 27	Instructor #3
1:00 - 2:00	Total, Dissolved, Suspended and Settleable Solids	14	Instructor #2
2:00 - 2:10	Break		
2:10 - 2:55	Laboratory		
	Total Solids	26	Instructor #3
	Suspended Solids	27	Instructor #1
3:00 - 3:30	Volatile Solids	14	Instructor #3
3:30 - 4:45	Elements of a Quality Assurance Program	5	Instructor #2
FRIDAY			
8:00 - 8:30	First Weighing of Suspended Solids	27	Instructors #1, 2, 3
	V METER ANALYSES		
8:30 - 9:15	Turbidity	15	Instructor #1
9:15 - 9:45	Group A - Weigh Total Solids	26	Instructor #1
	- Break		
	Group B - Break		
	- Weigh Total Solids	26	Instructor #1
9:45 - 10:30	Specific Conductance	16	Instructor #3
10:30 - 12:15	Laboratory		
10:30 - 11:20	Group A - Turbidity	28	Instructor #1
	Second Weighing of Solids	26, 27	Instructor #1
	Group B - Specific Conductance	29	Instructor #3
11:25 - 12:15	Group A - Specific Conductance	29	Instructor #3
	Group B - Turbidity	28	Instructor #1
	Second Weighing of Solids	26, 27	Instructor #1
12:15 - 1:00	Lunch		
1:00 - 1:30	Calculations and Class Discussion:		
	Specific Conductance		Instructor #3
	Solids Data		Instructor #1
	Turbidity Data		Instructor #1
1:30 - 2:15	Post-Course Evaluation		Course Coordinator
2:15 - 2:30	Course Closing		Course Coordinator

*Note: For actual course presentation, insert the name of the primary instructor opposite the title of his/her assignment. Assistant instructors are designated by ( ).

D. MILESTONES IN COURSE PLANNING AND PREPARATION

The following pages list major areas of course responsibilities in a chronological order to facilitate orderly and timely accomplishment. The table also serves as an example for assignment of these responsibilities to various staff members. It has been successfully used by NTOTC to conduct this course in the past.

The table headings are job titles associated with the listed tasks. A suggested staff is cited, including a laboratory assistant. It is recognized, however, that staff is often limited and one individual may serve in several of the defined roles. Having this summary according to an ideal situation should facilitate an equitable division of the required tasks among fewer persons.

Before using the milestones table, decisions must be made about the course content. It may be desirable to teach the approved test procedures for parameters that are not included in this package, but that are required locally to meet regulatory requirements. In that case, the table must be changed. Delete items identified for topics you omit and add the items needed for the topics you want to add.

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
5 TO 6 MONTHS BEFORE COURSE							
Determination of the need and decision to have course.	x						
Designation of Course Director and Course Secretary.	x						
Review responsibilities.		x					
Review responsibilities.			x				
Commit classroom and laboratory facilities.	x	x					
Develop and release Course Announcement including location, date, general statement of course content and training objectives.		x	x				
Prepare all forms and information sheets related to student registration procedures.		x	x				
Decide on staff members.	x	x					
4 TO 5 MONTHS BEFORE COURSE							
Receive, review, act upon Course Applications, continuing until course begins.		x	x				
Maintain records on deposition of each application, continuing through course.			x				
Inventory Instructor's Guides. Order needs.		x	x				

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
3 MONTHS BEFORE COURSE							
Commit all staff members who will participate in Course.	x	x		x	x	x	x
Develop Milestone Checklist for Course.		x	x				
Distribute copies to staff of Milestones, Instructor's Guide, Student Manual and any pertinent training resources.	x	x	x				
Review responsibilities.				x	x	x	x
Assign topics to Primary (P) and Assistant (A) Instructors:	x	x					
Sample Handling				P			
Compliance Methodology					P		
Laboratory Safety						P	
Analytical Techniques				P			
Volumetric Analysis					P		
pH				P			
Acidity and Alkalinity				A	P		
Precision					P		
Hardness						P	
Chlorine					P		
Phosphorus					A	P	
Spectrophotometer						P	
Accuracy					P		
Fluoride				P			
Nitrate and Nitrite				A	P		
Solids				A	P	A	
Quality Assurance					P		
Turbidity				P			
Specific Conductance						P	
Develop summary plan for course.		x	x	x	x	x	x
Inventory chemicals and laboratory equipment/supplies. List and commit lending sources. Order rest of needs.		x	x	x	x	x	x
Inventory classroom equipment/supplies. List and commit lending sources. Order rest of needs.		x	x				

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Inventory student training manuals. Order needs.		x	x				
2 MONTHS BEFORE COURSE							
Finalize Course Schedule (Agenda).		x	x	x	x	x	x
Request laboratory/classroom needs from lending sources.		x	x	x	x	x	x
Request training aids from lending sources.		x	x	x	x	x	
6 WEEKS BEFORE COURSE							
Check out operation of all major pieces of equipment.				x	x	x	x
Primary and Assistant Instructors go through laboratory procedures in student training manual, using IPWs to standardize instructions for students.				x	x	x	
1 MONTH BEFORE COURSE							
Summary (to date) to staff of registered students, continuing to course beginning.		x	x				
Check on progress of staff preparations for instruction, continuing through course.		x		x	x	x	
Prepare all administrative forms and materials needed for course presentation.		x	x				
Plan and rehearse classroom presentations using all required training aids. Finalize.				x	x	x	

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Obtain any duplicated instructional materials (data sheets, etc.).			x	x	x	x	x
Review summary of laboratory equipment and supply needs for expected number of students doing the selected procedures.		x					x
Clean all glassware required by students. Note any special requirements.							x
Reserve all specially-cleaned glassware or apparatus.							x
Assemble other student equipment and supplies.							x
2 WEEKS BEFORE COURSE							
Arrange for security of classroom and laboratory.		x	x				
Make reagents required by students EXCEPT those with specified, limited stability.				x	x	x	x
Make final arrangements to obtain required samples. Plan for any "synthetics."				x	x	x	x
Determine range of concentration of desired constituent(s) in samples for the course.				x	x	x	
Arrange for disposal of special test wastes.					x		x
(Continued)							

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Give Laboratory Assistant final list of equipment and supplies to be at each laboratory position. Discuss arrangement of shared equipment.				x	x	x	x
1 WEEK BEFORE COURSE							
Inform building food service of number of expected students and course lunch times (as appropriate).			x				
3 DAYS BEFORE COURSE							
Finalize seating arrangement for classroom.		x					
Assemble course materials in classroom (student manuals, administrative materials, etc.). Distribute as appropriate.		x	x				
Ready classroom instructional aids (boards, erasers, etc.)		x	x				x
Check out all classroom equipment (electrical systems, PA, projection equipment) and obtain back-up accessories (bulbs, etc.).		x					
COURSE OPENING							
Conduct opening exercises.		x					
Participate in course opening.	x		x	x	x	x	x
Complete any required student records, including roster.		x	x				
(Continued)							

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Prepare course certificates if needed at end of week.		X	X				
EVERY DAY OF COURSE							
Maintain general supervision of course.		X					
Prepare unstable reagents and/or samples on day of test.				X	X	X	X
Obtain samples for each test on day of test.				X	X	X	X
When assistant instructor, make any student evaluation records requested by the lead instructor.				X	X	X	
When primary instructor, compile evaluation record for each student.				X	X	X	
Keep any general records (e.g. attendance) as required to document successful course completion.		X		X	X	X	
Oversee disposal of special test wastes.					X		X

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
SECOND-LAST DAY OF COURSE							
Distribute course critique sheet to students.		x					
LAST DAY OF COURSE							
Check and sign course certificates if applicable		x					
Collect students' critique sheets.		x					
Conduct closing exercises.		x					
Participate in course closing.	x		x	x	x	x	x
Clean up classroom and laboratory.		x	x	x	x	x	x
WITHIN TWO WEEKS OF COURSE PRESENTATION							
Return or replace any borrowed classroom equipment/supplies.		x	x				
Return or replace any borrowed laboratory equipment/supplies.				x	x	x	x
Return or replace any borrowed training aids.		x	x	x	x	x	
Order repairs or replacements of own equipment used in course.		x	x	x	x	x	
Complete and file evaluation records on all students in predetermined area.			x	x	x	x	
See that all students have been appropriately notified of their degree of success in completing the course.		x	x				

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Participate in staff session on evaluation of course and recommendations for future offerings.	x	x	x	x	x	x	x
Prepare course summary/ evaluation report.		x					
Complete and file entire course records in mutually determined area.		x	x				

E. INSTRUCTIONAL RESOURCES

Most training institutions will make the fullest possible use of available instructional resources. The purpose of this section is to describe the nature, sources and availability of instructional resources suggested for use with this course.

1. Student Training Manual and Instructor's Guide for the course, "Inorganic Analyses in Water Quality Control Programs." Ordering information may be obtained from:

   U.S. Environmental Protection Agency
   National Training and Operational Technology Center
   Cincinnati, Ohio 45268
   (513) 684-7501

2. Slides, Slide/Tape Units, Overhead Transparencies and Videocassette (See IPW Sections VIII B and XI for a description). According to topic, these instructional training aids are:

a. Acidity and Alkalinity

   1) X-20: "Alkalinity", 28 assembled slides

b. Calcium and Magnesium Hardness

   1) X-22: "Hardness", 23 assembled slides

c. Chlorination and Chlorine Determinations

   1) X-21: "Chlorine", 36 assembled slides

   2) KEH-70: "Amperometric Determination of Total Chlorine in Wastewater" 11 minute 3/4" U-matic CCTV cassette produced by Kirkwood Community College.
   It is available for review and purchase from the College, 6301 Kirkwood Blvd., Cedar Rapids, Iowa 52406.

d. Determinate Error-Accuracy

   1) X-25: "Accuracy", 14 assembled slides

e. Elements of a Quality Assurance Program

   1) X-26: "Quality Assurance", 5 assembled slides

f. Fluoride

   1) XT-80: "Fluoride Analytical Procedures", 46 slides, 16 minute tape and script

g. Indeterminate Error-Precision

   1) X-24: "Precision", 20 assembled slides

h. Laboratory Safety Practices

   1) X-27: "Laboratory Safety", 40 assembled slides

i. Nitrate and Nitrite Nitrogen

   1) X-23: "Nitrate and Nitrite Nitrogen", 20 assembled slides

j. pH

   1) OT-11: "pH", 7 overhead transparencies

k. Phosphorus

   1) XT-44: "The Determination of Phosphorus", 53 slides, 14 minute tape and script

l. Sample Handling-Field Through Laboratory

   1) X-17: "Sample Handling", 8 assembled slides

m. Solids

   1) X-28: "Solids", 32 assembled slides

n. Specific Conductance

   1) X-29: "Specific Conductance", 15 assembled slides

o. Turbidity

   1) X-30: "Turbidity", 10 assembled slides

p. Use of a Spectrophotometer and Calibration Graphs

   1) X-8: "Use of a Spectrophotometer", 16 assembled slides

   2) X-9: "Calibration Graphs", 6 assembled slides

   3) XT-51: "Use of the Spectronic 20 Spectrophotometer", 35 slides, 8 minute tape and script

q. Volumetric Analysis

   1) X-18: "Volumetric Analysis", 30 assembled slides

All units described in 2. above are available on scheduled loan from NTOTC to institutions conducting this course. Requests should contain the information items on the "Request for Loan" form at the end of this section. Send requests to the National Training and Operational Technology Center at the address on page 4-1.

It is urged that materials desired from NTOTC for a specific course offering be requested in a single, consolidated communication. This will give greatest assurance of a well-coordinated response. Because these requests ordinarily will cover a number of different items, telephonic requests should not be made.

Requests should be timely. To assure effective delivery in time for use in the course, requests should be received at NTOTC at least 45 days prior to the course date. NTOTC will, in turn, make every effort to assure that the requested materials are delivered to the requesting institution several days prior to the start of the course in which they are to be used. This will permit review and practice by the instructional staff for the most effective use of such resources.

It is expected that all borrowed resources be returned to NTOTC within two weeks after completion of the course in which they are used.

With returned borrowed training resources, it is requested that the user provide NTOTC with an evaluation of the training resource(s) used. In this manner, the experience of users can be a factor in continuous improvements and responses to problems in using the resources. All reports on the use of such resources should include the number of students with whom the material was used.

3. Supportive References

   a. Manual: EPA-EMSL, "Methods for Chemical Analysis of Water and Wastes". This is the reference source for many of the methods presented in this course. Address requests for a copy to:

      U.S. Environmental Protection Agency
      Environmental Monitoring and Support Laboratory (EMSL)
      Cincinnati, Ohio 45268

   b. "Standard Methods for the Examination of Water and Wastewater" (14th edition), APHA, AWWA, WPCF. Available from Publication Office, American Public Health Association, Inc., 1015 18th Street, N. W., Washington, DC 20036.

   c. "Annual Book of Standards" Part 31, "Water", 1975. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

Note: Items 3. a., b., and c. are those currently cited by the Federal Registers for National Pollutant Discharge Elimination System (NPDES) and National Interim Primary Drinking Water Regulations (NIPDWR) (copies are in the Instructional Package Worksheet on Compliance Methodology). Contact your U.S. EPA Regional Quality Assurance Coordinator for pertinent Federal Registers issued after 8/80. (Call U.S. EPA, EMSL, at 513/684-7301, for the name and phone number of your Coordinator).

d. Catalog: "Water Quality Control Instructional Materials." This includes visual instructional units developed by NTOTC. Although not developed specifically for this course, several of the units are on course topics and might be useful supplementary material. Address requests for a copy to NTOTC at the address given on page 4-1.

4. Instructional Resources Already in Possession of the Training Institution

a) Many training organizations prefer to develop, or have developed, their own texts and audiovisual training resources.

REQUEST FOR LOAN
AUDIOVISUAL INSTRUCTIONAL UNIT

Title and Catalog No. ____________________________________

____

Intended Use: ____

____

Preferred Date of Use: ___________________________________

Alternate Date: ____

BORROWER'S NAME ____

Title ____

Organization ____

Address ____

____
(Zip)

Phone Number (include
Area Code): ______________________________

There is no charge for use of the Audiovisual Instructional Units. However, the *BORROWER* assumes financial responsibility for the value of all loaned equipment and instructional materials.

Unless special arrangements are made with the loaning office, units should be returned within two weeks. Return the unit by *REGISTERED, CERTIFIED* or *INSURED MAIL IMMEDIATELY* after use.

EPA-171 (Cin)
(8-74)

F. LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS

The consolidated list in this section is for overall planning purposes. It was compiled from the Instructional Package Worksheet (IPW) sections (part II of this manual) IX, "IPW Equipment and Supply Requirements" and X, "IPW Reagent Requirements." Sections IX and X can also be used on a day-to-day basis during the course to prepare for the laboratory exercises.

The first column of this consolidated list contains a description of the item as found in commercial catalogs.

The second column identifies the tests utilizing the equipment. At the beginning of the table, there is a key to the abbreviations used in this column.

The third column lists the minimum quantity per laboratory station (no more than two persons) required for each test. In many cases, multiple use of the item is required to carry out the procedure. The quantities are based on the assignment stated in section IX of the related IPW. For convenience, a list of the assignments for each topic can be found as part of the key for abbreviations at the beginning of the table. If an Instructor chooses a different assignment for a topic, some quantities must be changed accordingly. As noted, numbers represent minimum quantities. It is strongly recommended that Instructors provide surplus equipment and additional supplies ready for use in case of need. Many Instructors plan for a margin of at least 10% of extra supplies to provide for student errors, planning miscalculations, or other unforeseen events.

The fourth and final column contains remarks that may be useful when deciding on class needs or when ordering equipment.

Before using the list, decisions must be made about the course content. It may be desirable to teach the approved test procedures for parameters that are not included in this package, but that are required locally to meet regulatory requirements. In that case, the list must be changed. Delete items identified for topics you omit and add the items needed for the topics you want to add.

This list can be of great value in pre-course planning, to determine the availability of needed equipment and supplies, and to take action to provide needed resources. Further, this list can be of vital importance when planning for courses to be conducted in field locations. Copies of the list in the hands of the Course Coordinator and a representative of the host organization can be used to determine who will provide needed resources on an item-by-item basis. When the responsibility is assigned/accepted, this can be annotated in the "remarks" column on the copy in the hands of the Course Coordinator and the copy of the representative of the host organization. Each can then use the annotated equipment and supply list as a checklist for carrying out his own agreed-upon responsibilities in preparing for the course.

Following is the key for abbreviations used for course topics in the table. The second column lists the laboratory assignment for each station as given in the corresponding IPW in Part II of this guide. (There should be no more than two persons at a station.) The quantity given for each laboratory item in the table was calculated according to the IPW assignment. If you change the assignment, adjust the quantities accordingly.

ABBREVIATIONS FOR TESTS	IPW ASSIGNMENTS (per station)
A/A Acidity and Alkalinity, Titration	1 sample for acidity 2 samples for alkalinity
Cl Chlorine, Amperometric (Back) Titration	1 sample for two titrations
F Fluoride, SPADNS and Electrode with Specific Ion Meter	4 samples by each method
H Hardness, Titration	tap water 1 standard 1 effluent sample (or synthetic)
N/N Nitrate and/or Nitrite, Cadmium Reduction	Prepare and Activate a column Zero standard plus 1 standard or sample for nitrate. Same for nitrite.
P Total Phosphorus, Digestion and Reduction	2 standards (zero + 1 other) 1 sample
S Solids-Total and Suspended, Gravimetric	1 influent sample for total solids 1 influent and 1 effluent sample for suspended solids.
SC Specific Conductance, Meter	Determine cell constant for 3 ranges 3 samples
T Turbidity, Nephelometer	Dilute a stock solution to make 3 or 4 standards. 2 samples (tap water + 1 synthetic)

LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS
(Compiled from Section IX in the IPW's)

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
APRONS, laboratory (plastic acceptable)	All	See Remarks	One per person
BALANCES, analytical, 0.1 mg sensitivity at a load of 200 g, with Instruction Manuals	S	See Remarks	1 per 3 stations
BEAKERS, glass, 50 ml	A/A	See Remarks	2 in hood for H_2O_2
BEAKERS, glass, 100 ml	A/A P	4 1	
BEAKERS, glass, 150 ml	A/A H N/N	3 1 2	H - If two electrodes are used for pH, a second beaker is needed.
BEAKERS, plastic, 150 ml	F	6	
BEAKERS, glass, 250 ml	H N/N	1 2	
BEAKERS, glass, 400 ml	N/N SC	1 7	
BEAKERS, glass, 600 ml	SC	1	
BOILING BEADS, glass, 5mm	A/A P	See Remarks See Remarks	In one supply container. Need: A/A - 3 each station P - 12 each station
BOTTLES, glass or plastic, about 2.5 liter for Cd rinsings. Used acid bottles serve well.	N/N	1	

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
BRUSHES, balance	S	See Remarks	1 by each BALANCE
BURETS, 25 ml, 0.1 ml graduations, teflon stopcock plug preferred	A/A H	1 1	
CLAMPS, buret for titration stand, to hold two BURETS	A/A H N/N	1 1 1	
CLAMPS, screw to regulate flow through 4 cm ID rubber TUBING	N/N	1	
CLAMP, pinchcock for 4 cm ID rubber TUBING	N/N	1	
CONDUCTIVITY METERS, Wheatstone bridge with cell(s) to measure conductance ranging from 15 to 13,000 µ mhos/cm @ 25°C. Each should have an accessory or means to check the resistance.	SC	1	Conducting the lab session twice halves the number needed.
CONTAINERS for instrument cell wastes, about 2 liters	Cl N/N F P H T	See Remarks	All - 1 per 2 stations
CRUCIBLES, Gooch, porcelain, 25 ml capacity	S	1	
CRUCIBLE HOLDERS, Walter, for 25 ml crucible to fit FILTERING ASSEMBLY	S	1	
CYLINDERS, graduated, 10 ml	H	1	
CYLINDERS, graduated, 25 ml	H S	1 1	
CYLINDERS, graduated 50 ml	N/N	1	

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
CYLINDERS, graduated, 100 ml	N/N S	1	
CYLINDERS, graduated, 200 ml	Cl	1	
DESICCATORS, with active desiccant	S	See Remarks	Capacity for 1-25 ml GOOCH CRUCIBLE, 1-150 ml EVAPORATING DISH and 1-6 cm WATCH GLASS or PLANCHET per station
EVAPORATING DISHES, 150 ml, porcelain	S	1	
FILTERING ASSEMBLIES for 10 ml, 50 ml, and 100 ml volumes.	P S	1 1	P - Filtrate to be transferred S - Filter support should be ∿ 5cm diameter for influent filtration
FILTER DISCS, phosphorus-free, 0.45 micron pore size, Gelman GA-6 or equivalent, to fit FILTERING ASSEMBLIES	P	3	
FILTER DISCS, glass fiber, no organic binder, Reeve Angel 934AH, Gelman type A/E, Millipore AP-40, Whatman GF/C or equivalent. Diameter ∿ 5cm for FILTERING ASSEMBLY and also ∿ 2.1 cm for CRUCIBLE	S	1 Each Size	
FILTER PAPER, fluted, about No. 12 to fit 14 cm FUNNEL for Cd rinsings	N/N	1 piece	
FLASKS, Erlenmeyer, wide mouth, 125 ml	F H P	6 See Remarks 3	H - 1 If two electrodes are used for pH, use 1-150 ml BEAKER instead
FLASKS, Erlenmeyer, wide mouth, 250 ml	N/N	4	
FLASKS, volumetric, 50 ml, with glass stoppers	P	3	
FLASKS, volumetric, 100 ml with glass stoppers	N/N T	3 4	
FORCEPS, pairs, blunt-tipped, to manipulate FILTER DISCS	P S	1 1	

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
FUNNELS, short stem, diameter about 5 cm to fit neck of 50 ml VOLUMETRIC FLASK and 25 or 50 ml BURET	A/A H P	1 1 1	
FUNNELS, medium stem, diameter about 14 cm to fit neck of old BOTTLES for Cd rinsings	N/N	1	
GLASSES, pairs of safety	All	See Remarks	One per person
GLASS WOOL, small wad for end of 100 ml cut-off PIPET	N/N	1	
HOT PLATES with continuous settings, not just "low", "medium", and "high".	A/A P	See Remarks See Remarks	A/A: 1-150 ml BEAKER per station P: 3-125 ml ERLENMEYER FLASKS per station
OVEN, hot air, to give uniform temperatures and with thermometer to register accurately in range of 103°-105°C.	S	See Remarks	Capacity for 1-25 ml Gooch CRUCIBLE, 1-150 ml EVAPORATING DISH and 1-6cm WATCH GLASS or PLANCHET per station
OVEN, a second oven as described above or a steam bath for use at 98°C	S	See Remarks	Capacity for 1-150 ml EVAPORATING DISH per station
PAPER CLIPS	N/N	1	
PENCILS, WAX MARKING	All	1	
pH METERS, ELECTROMETRIC, accurate to at least 0.1 pH unit, each with electrode(s)	A/A H N/N P	1 See Remarks See Remarks 1	Conducting the lab session twice halves the total number needed. H - 1 per 2 stations N/N - 1 per 2 stations
PIPETS, dropping (medicine droppers) with bulb about 1 ml	A/A	See Remarks	2 in hood for H_2O_2
PIPETS, measuring, glass, 1 ml graduated at 0.1 ml	Cl	1	
PIPETS, measuring (Mohr), glass, 10 ml	N/N P	See Remarks 1	N/N - 1 for each bottle of color reagent

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
PIPETS, volumetric transfer, 1 ml	N/N P	See Remarks See Remarks	N/N - 2 per 4 stations P - 1 per 6 stations
PIPETS, volumetric transfer, 2 ml	N/N	See Remarks	2 per 4 stations
PIPETS, volumetric transfer, 3 ml	P	See Remarks	1 per 6 stations
PIPETS, volumetric transfer, 5 ml	Cl N/N P	1 See Remarks See Remarks	Cl - may need graduated pipet for titrator N/N - 2 per 4 stations P - 1 per 6 stations
PIPETS, volumetric transfer, 10 ml	F N/N P	3 See Remarks See Remarks	N/N - 2 per 4 stations P - 1 per 6 stations
PIPETS, volumetric transfer, 20 ml	P	See Remarks	P - 1 per 6 stations
PIPETS, volumetric transfer, 25 ml	H N/N	1 1	
PIPETS, volumetric transfer, 50 ml	A/A F N/N P	See Remarks 1 1-3 1-2	A/A - 1 for each bottle of sample N/N - 3 if possible P - 2nd pipet - 1 per 6 stations
PIPETS, volumetric transfer, 100 ml	T	4	
PIPETS, volumetric transfer, 100 ml with top and tip cut off for reduction column	N/N	1	REDUCTION COLUMNS can be purchased.
PIPETS BULBS, large with opening shaped to fit variety of PIPET sizes	A/A P F T H	1	
PLANCHETS, aluminum or stainless steel, about 6 cm diameter	S	1	WATCH GLASSES may be used instead.
PROPIPET BULBS	Cl N/N	1 1	

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
REAGENT SPOONS, 0.4 gram	P	1	
REDUCTION COLUMNS	N/N	1	Can be purchased or made. See "PIPETS, vol. transfer, 100 ml, etc"
SAFETY GLASSES	All	See Remarks	One pair for each person
SIEVES, 60 mesh	N/N	1	
SPATULAS	A/A Cl H N/N P	See Remarks	A/A and P: One in hood for transferring BOILING BEADS from common supply container. Other tests: One at each station
SPECIFIC ION METERS, each with a fluoride ion activity electrode and a reference electrode or with a combination fluoride electrode	F	1	Conducting the lab session twice and then alternating with SPADNS test quarters the total number needed.
SPECTROPHOTOMETERS, for use at 570, 540, 880 (or 650) nm, providing a light path of at least 1 cm	F N/N P	1 See Remarks See Remarks	F - 570 nm See "Remarks" for SPECIFIC ION METERS about number needed. N/N - 540 nm One per 3 stations P - 880 or 650 nm One per 3 stations, special filter & phototube maybe needed.
SPECTROPHOTOMETER CELLS, 1 cm for instruments on hand	F N/N P	See Remarks	One for each instrument If matched sets of cells are available, then one set per instrument
STANDS, titration with porcelain bases	A/A H N/N	1	
STIRRING APPARATUS, MAGNETIC	A/A Cl H F		Chlorine TITRATOR may include a stirring mechanism
STOP WATCHES, or timing devices	F	1	
THERMOMETERS	A/A F H N/N P SC	See Remarks	All - 1 for each pH METER or SC METER
TISSUES, small, soft, in boxes	Cl S SC	See Remarks	Cl - 1 box per 2 instruments S - 1 box per desiccator plus 1 box per balance SC - 1 box per 2 instruments

DESCRIPTION	TEST(S)	QUANTITY PER STATION	REMARKS
TITRATORS, amperometric with cell and any accessories, e.g., pipets.	Cl	1	Conducting the lab session twice halves the total number needed.
TONGS, crucible, pairs	S A/A	1	
TUBING, soft rubber, 4 cm ID, 7.5 cm length	N/N	1	
TURBIDIMETERS, Nephelometers that measure light scattered at right angles to the path of the incident light, with cell and any standard accessories.	T	1	Conducting the lab session twice halves the number needed.
VACUUM SOURCES: preferably a central service. An electric vacuum pump assembly with suitable hoses, water traps and shut off valves and with capability of drawing 15 inches of mercury can be used.	S	1	
WASH BOTTLES, plastic squeeze type, 500 ml	All	1	
WATCH GLASSES, about 6 cm diameter	S	1	PLANCHETS may be used instead.
WATER STILL to produce water free of the constituent sought in each test.	All	See Remarks	One, unless ion-free water is purchased.

LABORATORY REAGENT REQUIREMENTS
Compiled from Section X in the IPW's
ALL QUANTITIES ARE MINIMUMS

DESCRIPTION	QUANTITY PER STATION	REMARKS
ACIDITY and ALKALINITY	See Remarks	For DETAILS OF REAGENT PREPARATIONS, see 14th edition, "Standard Methods", pages 276 and 279 (or the method cited if a later edition is approved).
Buffer, pH 4.X - 1000 ml 10.12 g potassium biphthalate	75 ml	Used to standardize the pH meter for these tests and also for NITRATE/NITRITE and PHOSPHORUS. Commercial powders or tested solutions may be used.
Buffer, pH 6.X - 1000 ml 3.388 g potassium dihydrogen phosphate 3.533 g disodium hydrogen phosphate	150 ml	Used to standardize the pH meter for these tests and also for HARDNESS, NITRATE/NITRITE and PHOSPHORUS. Commercial powders or tested solutions may be used.
Buffer, pH 9.X - 1000 ml 3.80 g sodium borate decahydrate (borax)	75 ml	Used to standardize the pH meter for these tests and also for HARDNESS. Commercial powders or tested solutions may be used.
Hydrogen peroxide, 30 % solution	5 drops	
Potassium biphthalate - 1000 ml 10.0 g	See Remarks	Used to standardize 0.1 N sodium hydroxide
Sodium carbonate - 1000 ml 2.5 g	See Remarks	Used to standardize 0.1 N sulfuric or hydrochloric acid. Also used for SAMPLE for Alkalinity.
Sodium hydroxide, 0.1N-1000 ml 11 g	See Remarks	Used to prepare 0.020 N sodium hydroxide
Sodium hydroxide, 0.020 N - 1000 ml 200 ml 0.1 N sodium hydroxide	15 ml	Also used for SAMPLE for Alkalinity
Sulfuric acid, 0.1 N - 1000 ml 3.0 ml concentrated sulfuric acid	See Remarks	Used to prepare 0.020 N standard acid. You can use 0.1 N hydrochloric acid instead of sulfuric. Prepare by diluting 8.3 ml HCl to 1 liter.
Sulfuric acid, 0.020 N - 1000 ml 200 ml 0.1 N sulfuric acid	25 ml	-Can prepare 200 ml 0.020 N hydrochloric acid the same way. -Also used for SAMPLE for Acidity
Sample for acidity - 1000 ml 300 ml 0.020 N sulfuric or hydrochloric acid	50 ml	Wastewater treatment plant influents or effluents known to contain acids may be used.

DESCRIPTION	QUANTITY PER STATION	REMARKS
Sample for alkalinity - 1000 ml 300 ml 0.020 N sodium carbonate	50 ml	Wastewater treatment plant influents or effluents known to contain alkalinity may be used.
Sample for alkalinity - 1000 ml 200 ml 0.020 N sodium hydroxide	50 ml	Wastewater treatment plant influents or effluents know to contain alkalinity may be used.
CHLORINE, Amperometric (Back) Titration	See Remarks	For DETAILS OF REAGENT PREPARATION, see 14th edition "Standard Methods", page 318 (or the method cited if a later edition is approved).
Arsenite solution, 0.1 N - 1000 ml 4.95 g arsenic trioxide 15 g sodium hydroxide carbon dioxide to saturate 250 ml sol.	See Remarks	Used to standardize 0.1 N iodine solution
Buffer, pH 4.0 - 1000 ml 243 g sodium acetate trihydrate 480 g glacial acetic acid	8 ml	This reagent can be purchased.
Hydrochloric acid - 5 to 10 drops	See Remarks	Used in standardizing 0.1 N iodine solution
Iodine solution, 0.1 N - 1000 ml 40 g potassium iodide 13 g resublimed iodine	See Remarks	Used to prepare 0.0282 N iodine solution The 0.1 N iodine solution can be purchased
Iodine solution, 0.0282 N - 1000 ml 25 g potassium iodide ~ 282 ml of solution prepared to be 0.1 N iodine	2 ml	Used as a titrant in the test and also to standardize phenylarsine oxide solution. Actual amount of 0.1 N iodine depends on results from its standardization.
Phenylarsine oxide, 0.00564 N - 1000 ml 0.8 g phenylarsine oxide 1.8 g sodium hydroxide 1 + 1 hydrochloric acid (to adjust pH) 0.05 g potassium iodide	10 ml	This reagent can be purchased. Must use 0.0282 N iodine solution to finalize this and then to check the final normality.

DESCRIPTION	QUANTITY PER STATION	REMARKS
Potassium iodide crystals	2 g	Also used in preparing some of the above reagents. One ml of a solution of 5 parts KI in 95 parts distilled water can be used instead of 2 g of crystals.
Sodium chloride, U.S.P., pellets	20 g	Electrolyte for cell
Starch indicator ~ 1 g soluble powder	See Remarks	Used to standardize 0.1 N iodine and 0.00564 N phenylarsine oxide
Sample	400 ml	UNSTABLE - Prepare just prior to use, about 3 mg/liter. Dilute a chlorine solution or a hypochlorite bleach. A chlorinated plant effluent may be used.
FLUORIDE, SPADNS and Electrode	See Remarks	For DETAILS OF REAGENT PREPARATION, see 14th edition, "Standard Methods", and page 393 and 392 (or the method cited if a later edition is approved).
Acid Zirconyl - SPADNS reagent - 1000 ml 500 ml SPADNS solution 500 ml Zirconyl - acid reagent	75 ml	This combined reagent is stable for at least two years. SPADNS solution and Zirconyl-acid reagent are listed below.
Fluoride stock solution - 1000 ml 0.2210 g anhydrous sodium fluoride	See Remarks	Used to make Fluoride standard solutions and four samples.
Fluoride standard, 1 mg - 1000 ml 10 ml stock solution	100 ml	UNSTABLE - Prepare just prior to use.
Fluoride standard, 2 mg - 1000 ml 20 ml stock solution	100 ml	Required if a pH meter is used.
Fluoride standard, 10 mg - 1000 ml 100 ml stock solution	60 ml	
SPADNS solution - 1000 ml 1.916g SPADNS, sodium 2-(para-sulfophenylazo)-1,8-dihydroxy-3,6-naphthalene disulfonate	See Remarks	This solution is stable indefinitely if protected from direct sunlight. Used to make Acid Zirconyl-SPADNS reagent.

DESCRIPTION	QUANTITY PER STATION	REMARKS
TISAB - 1000 ml 57 ml glacial acetic acid 58 g sodium chloride 4.0 g 1, 2 cyclohexylene dinitrilo tetraacetic acid (CDTA) ∿ 30 g sodium hydroxide	300 ml*	This reagent can be purchased. *400 ml is required if a pH meter is used.
Zirconyl - acid reagent - 1000 ml 0.266 g zirconyl chloride octahydrate 700 ml concentrated hydrochloric acid	See Remarks	Used to make Acid Zirconyl - SPADNS reagent.
Sample #1 - 1000 ml 5.5 ml fluoride stock solution	100 ml	
Sample #2 - 1000 ml 10.5 ml fluoride stock solution	100 ml	
Sample #3 - 1000 ml 7.5 ml fluoride stock solution 0.4437 g anhydrous sodium sulfate	100 ml	
Sample #4 - 1000 ml 9.5 ml fluoride stock solution 52.752 mg aluminum potassium sulfate (also called potassium alum) with ·12 H_2O	100 ml	
HARDNESS, titration	See Remarks	For DETAILS OF REAGENT PREPARATION, see 14th edition, "Standard Methods", page 203 (or the method cited if a later edition is approved).
Buffer, pH 6.X	100 ml	See this item in ACIDITY and ALKALINITY section.
Buffer, pH 9.X	100 ml	See this item in ACIDITY and ALKALINITY section.
Buffer, test reagent - 1000 ml 67.6 g ammonium chloride 572 ml concentrated ammonium hydroxide 4.716 g EDTA (also listed below) 3.120 g magnesium sulfate ·7 H_2O OR 2.576 g magnesium chloride ·6 H_2O	6 ml	Store in tightly-stoppered plastic or resistant-glass container. Do not store more than a month's supply in a frequently opened container. See "Standard Methods" regarding alternate "odorless buffers."
Calcium carbonate - 1000 ml 1.000 g anhydrous calcium carbonate 1+1 hydrochloric acid to dissolve calcium carbonate and adjust pH 3N(1+4) ammonium hydroxide for pH 3 drops methyl red indicator	25 ml	Also used to standardize EDTA.

DESCRIPTION	QUANTITY PER STATION	REMARKS
EDTA, 0.01 M - 1000 ml 3.723 g disodium ethylenediamine tetra-acetate dihydrate	75 ml	
Indicator 0.5 g Eriochrome Black T 100 g sodium chloride	0.6 g	See "Standard Methods" for commercial equivalents.
Inhibitor See Remarks		This is usually not required. See "Standard Methods" for discussion and choices.
Sample - 1000 ml 10 ml calcium carbonate solution	25 ml	A wastewater treatment plant sample may be used if it will give positive hardness results.
NITRATE/NITRITE, Cadmium Reduction	See Remarks	For DETAILS OF REAGENT PREPARATIONS, see 1974 EPA, "Methods for Chemical Analysis", page 202 (or the method cited if a later edition is approved)
Ammonium chloride - EDTA, conc. - 1000 ml 1.7 g disodium ethylenediamine tetra-acetate 13g ammonium chloride conc. ammonium hydroxide to adjust pH	610 ml	400 ml is used for the testing of the assigned (4) solutions. The additional 210 ml is used to prepare 350 ml dilute solution for preparing one column.
Ammonium chloride - EDTA, dilute - 1000 ml 600 ml concentrated solution	350 ml	
Ammonium hydroxide, concentrated	∿ 1 ml	For pH adjustment of sample
Buffer, pH 4.X	100 ml	See this item in ACIDITY and ALKALINITY section.
Buffer, pH 6.X	100 ml	See this item in ACIDITY and ALKALINITY section.
Cadmium, granulated 40-60 mesh	∿ 20 g	Can be purchased
Color reagent - 1000 ml 10 g sulfanilamide 1 g N (1-naphthyl)-ethylenediamine dihydrochloride 100 ml concentrated phosphoric acid	8 ml	

DESCRIPTION	QUANTITY PER STATION	REMARKS
Copper sulfate, 2% - 1000 ml 20 g copper sulfate pentahydrate	200 ml	
Hydrochloric acid, concentrated	~ 1 ml	For pH adjustment
Hydrochloric acid, 6N-1000 ml 500 ml concentrated hydrochloric acid	60 ml	
Potassium nitrate stock - 1000 ml 7.218 g potassium nitrate 2 ml chloroform	See Remarks	Used to prepare activation and standard solutions. This solution is stable for at least six months, kept under refrigeration.
Potassium nitrate activation solution - 1000 ml 1 ml stock solution	25 ml	UNSTABLE - prepare just before use.
Potassium nitrate standard - 1000 ml 10 ml stock solution (for 1 cm cells)	15 ml	Also used to prepare sample UNSTABLE - prepare just use.
Potassium nitrite stock - 1000 ml 6.072 g potassium nitrite 2 ml chloroform	See Remarks	Used to prepare standard solution. This solution is stable for about three months, kept under refrigeration.
Potassium nitrite standard - 1000 ml 10 ml stock solution (for 1 cm cells)	15 ml	Also used to prepare sample. UNSTABLE - prepare just before use.
Sample - 1000 ml 40 ml nitrate standard 40 ml nitrite standard	50 ml	UNSTABLE - prepare just before use. A wastewater treatment plant sample can be used. This should contain both nitrate and nitrite.
PHOSPHORUS, Digestion & Reduction	See Remarks	For DETAILS OF REAGENT PREPARATIONS, see 1974 EPA, "Methods for Chemical Analysis", page 252 (or the method cited if a later edition is approved).
Ammonium persulfate	1.2 g	
Buffer, pH 4.X	100 ml	See this item in ACIDITY and ALKALINITY section
Buffer, pH 6.X	100 ml	See this item in ACIDITY and ALKALINITY section

DESCRIPTION	QUANTITY PER STATION	REMARKS
Combined Reagent - 1000 ml 70 ml sulfuric acid, conc., diluted to 500 ml 0.1372 g antimony potassium tartrate in sol. 6 g ammonium molybdate in solution 5.28 g ascorbic acid in solution	24 ml	LIMITED STABILITY - Combine the four solutions just before use. Follow all preparation instructions carefully.
Potassium acid phosphate stock - 1000 ml 0.2197 g potassium dihydrogen phosphate	See Remarks	Used to prepare standard solution and sample.
Potassium acid phosphate standard - 1000 ml 20 ml stock solution (for 1 cm cells)	70 ml	UNSTABLE - Prepare just before use.
Sodium hydroxide, 10N-1000 ml 400 g sodium hydroxide	~ 10 ml	For pH adjustment
Sodium hydroxide 0.1N-1000 ml 4 g sodium hydroxide	~ 5 ml	For pH adjustment
Sulfuric acid, 11N-1000 ml 310 ml concentrated sulfuric acid	~ 4 ml	For digestion and for treating sample
Sulfuric acid, 0.11N-1000 ml 3.1 ml concentrated sulfuric acid	~ 5 ml	For pH adjustment
Sample - 1000 ml 10 ml stock solution	50 ml	UNSTABLE - Prepare just before use. A wastewater sample containing phosphorus may be used.
SOLIDS, Total and Suspended	See Remarks	No reagents are needed for these gravimetric tests.
Sample, plant influent 25 to 200 mg total or suspended residue	~ 200 ml	If a treatment plant sample is not available, a quality control sample of suspended solids could be used for both tests. EPA-EMSL, Cincinnati, Ohio 45268 is one supplier.
Sample, plant effluent 25 to 200 mg suspended residue	~ 100 ml	See above Remarks for alternative.
SPECIFIC CONDUCTANCE, meter	See Remarks	For DETAILS OF REAGENT PREPARATIONS, see 14th edition, "Standard Methods", page 74 (or the method cited if a later edition is approved).

DESCRIPTION	QUANTITY PER STATION	REMARKS
Potassium chloride stock, 1N-1000 ml 74.56 g potassium chloride	See Remarks	Used to prepare two standards and Sample A.
Potassium chloride, 0.1N-1000 ml 100 ml 1N solution	400 ml	Also used to prepare 0.001 N solution and Sample B.
Potassium chloride, 0.01N-1000 ml 10 ml 1N solution	400 ml	Also used to prepare 0.0001N solution and Sample C.
Potassium chloride, 0.001N-1000 ml 10 ml 0.1N solution	400 ml	
Potassium chloride, 0.0001N-1000 ml 10 ml 0.01N solution	400 ml	
Sample A - 1000 ml 10 to 100 ml 1N solution	400 ml	A water sample with measurable conductivity may be used.
Sample B - 1000 ml 10-100 ml 0.1N solution	400 ml	A water sample with measurable conductivity may be used.
Sample C - 1000 ml 10-100 ml 0.01N solution	400 ml	A water sample with measurable conductivity may be used.
TURBIDITY, Nephelometer	See Remarks	For DETAILS OF REAGENT PREPARATIONS, see 1974 EPA, "Methods for Chemical Analysis", page 296 (or the method cited if a later edition is approved).
Formazin stock, 400 NTU-1000 ml 1 g hydrazine sulfate 10 g hexamethylenetetramine	10 ml	The stock is a mixture of solutions of the two chemicals listed. Also used to prepare Formazin standard
Formazin standard, 40 NTU-1000 ml 100 ml Formazin stock	See Remarks	Used to make sample.
Manufacturer's standards	See Remarks	As supplied to standardize the meter.
Sample One - 1000 ml 5 ml 40 NTU standard	50 ml	A water sample with measurable turbidity may be used.
Sample Two - Tap Water	50 ml	Another water sample with measurable turbidity may be used.

PART II - INSTRUCTIONAL PACKAGE WORKSHEETS

The Worksheets are for guidance to the Instructor who develops the subject matter covered in the course. These Worksheets are not scripts. The Instructor will need to make extensive and detailed preparation in order to perform the assigned tasks effectively and efficiently. The Instructional Package Worksheets do provide a perspective of each analytical procedure, the learning achievement levels the students should attain, available audiovisual and other instructional resources, and an example course of action in pre-course preparation and classroom/laboratory instruction. The Instructor is free to modify the Worksheets to meet individual needs. It should be noted however, that associated additions and deletions will then be required in Sections V through XI of the Worksheet, and in Sections E and F of Part I.

Application of these Instructional Packages will help the Instructor to reduce the time required for planning and organizing a strategy of preparation and instruction. However, time and effort are required for physical preparations for classroom and laboratory instruction; time and effort are required for rehearsals of Instructor performance in classroom and laboratory. These requirements never can be met by such an Instructor's Guide as this; ultimately the Instructor is the key person in assuring that the student acquires the needed knowledge and skills.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Course Objectives - Introductions

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 15 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To give overview of course and allow class to begin to know each other.

V. ENTRY LEVEL BEHAVIOR:

A. Application and acceptance as a participant in the course.

B. Registration forms and details have been completed.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will have an overview of the course schedule, be able to use the training manual and correlate it to the agenda, and will know the requirements for successful completion of the course as stipulated for earning continuing education units. He/she will also know more about the other members of the class.

B. Conditions: He/she will be provided with a course schedule, a copy of the training manual, and a short discussion by the Course Director.

C. Accepted Performance: Attention during the discussion and use of the provided materials as directed.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. The course schedule

2. The training manual

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Prepare a summary of the employers of the participants.

2. Review the requirements you have established for award of continuing education units for this course, if applicable. At NTOTC, we awarded 2.5 CEU's (1500 minutes contact time) with the following requirements:

        a. 95% attendance
        b. Satisfactory participation in class and laboratory sessions as judged by the course instructors
        c. A score of 70% or more on the post-course evaluation if the pre-course score on the same test was less than 50%. Pre-course scores of 50% or higher are to improve by at least 20% to a maximum requirement of 90%. Any exceptions to this standard must be documented in the course file.
    3. Duplicate copies of the course schedule for each student.
    4. Secure a copy of the training manual for each student.

B. Sequencing:

1. Welcome
2. Summary of employers of class participants. Special welcome to people from other countries.
3. Brief overview of course topics using the beginning pages in the training manual.
    a. Use page with course description. Restate the course objectives including the list of topics in the final paragraph.
    b. Contents page - location of lecture and laboratory outlines.
4. Agenda
    a. Note topics and manual outline numbers.
    b. Note breaks and lunch times. They schedule own break during laboratory sessions.
    c. Note time course ends.
    d. General format is lecture, laboratory, follow-up discussion for each topic.
5. Scan outlines before class, especially laboratory procedures.
6. Explain requirements for earning continuing education units for course (if applicable).
7. Have each person rise and give name, employer, and principle job responsibilities.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For each student:

1. One copy of the training manual.
2. One copy of the course schedule.

X. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. None

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Pre-Course - Post-Course Evaluation

II. UNIT OF INSTRUCTION: Summary of NTOTC Use of the Test

III. ESTIMATED TIME: 75 minutes (30 + 45)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: There is an "Accepted Performance" standard stated in the "Instructional Objective" for each subject in this course (VI C. in each IPW). It states the mode of evaluation used at NTOTC for that subject and includes the requirement that the participant must correctly answer 70% of the items on the subject in the post-course evaluation. Thus a minimum final score of 70% on the post-test is required. To earn continuing education units for the entire course (2.5 CEU's), NTOTC also requires evidence of increased knowledge by an increase of 20 points or more (to a maximum requirement of 90%) in the score for the same test taken before and after participating in the course.

The test contains items for every topic in the course. You may find these items useful even if you prefer to administer a written test for each topic as it is completed. You can compile topical tests using these items plus your own questions to obtain a more comprehensive evaluation of achievement regarding the individual subjects. (You may need to increase the time allotments given in the NTOTC course schedule for this additional evaluation activity).

V. ENTRY LEVEL BEHAVIOR:

A. Pre-Course Evaluation

1. Is a chemist or chemistry technician

2. Has the prerequisites to participate in the course

B. Post-Course Evaluation

1. Attended 95% of the course

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will answer the same set of test questions before and after participating in the course to fulfill the requirement for award of 2.5 continuing education units for the course.

B. Conditions: He/she will be given the test and 30 minutes.

C. Accepted Performance: An initial grade of less than 50 should increase to 70 or more. An initial grade of 50 or more should increase 20 points with 90 as a maximum required score.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. A set of questions on main points presented for each topic on the NTOTC course schedule, an answer sheet, scoring information and a key are at the end of this IPW.

2. An example letter sent to participants who attended 95% of the course but are not awarded CEU's is at the end of this IPW.

3. An example letter sent to participants who did not attend 95% of the course so are not awarded CEU's or a course certificate is at the end of this IPW.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Duplicate one copy of the questions to be used and two copies of the answer sheet for each student.

B. Sequencing:

1. Before the course topics are presented - 30 minutes

   a. If the "Instructional Objective" (VI above) has not been presented to the participants (in the preceding "Course Objectives" IPW), explain it now.

   b. Distribute one copy of questions and one answer sheet to each participant.

   c. Go over the directions. Emphasize that one should not write anything on the question sheets.

   d. Allow the rest of 30 minutes for each to complete the answer sheet.

   e. Collect all distributed materials. Make sure that each answer sheet has the name and date recorded.

2. Outside of class:

   a. Check all question sheets to be sure none have been marked in any way. Reserve for future use.

   b. Correct the answer sheets and assign scores.

   c. The results for groups of items are indicators of each participant's entry level knowledge about each topic.

3. After the course topics are presented - 30 minutes

   a. Distribute the same question sheets and one blank answer sheet to each participant

b. Repeat the procedures in 1. c. through e. above

4. After collecting completed surveys - 15 minutes

   a. Use acetates of the question sheets with circled answers to go through the test questions as a summary of course content.

5. Outside of class:

   a. Check all question sheets to be sure none have been marked in any way. Reserve the clean copies for future use.

   b. Correct the answer sheets and assign scores.

   c. For each participant, record the pre-course and post-course scores.

   d. Use the "Accepted Performance" standard to determine which participants have earned the CEU's for the course.

   e. Take appropriate action for each student regarding this requirement for award of continuing education units. At NTOTC, the action for those earning the CEU's is to make arrangements for recording the award on the person's course certificate. Those who attended the entire course, but who did not earn CEU's, receive a course certificate and a letter documenting why the CEU's are not recorded on the certificate. (An example of the letter is at the end of this IPW).

   f. Inspect the answer sheets, item-by-item. A tally of the errors per item can serve as an evaluation of the instruction given on the topic involved, as well as of the participants' mastery of the topic of the item (or group of items). Make a note if any changes in the instructional approach for topics should be planned for future courses.

   g. File the scored answer sheets with any other evaluation information about the students.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For Each Student:

1. One copy of the test questions and two copies of the answer sheet for each.

X. IPW REAGENT REQUIREMENTS:

A. None

INORGANIC ANALYSES IN WATER QUALITY PROGRAMS

EVALUATION

- Please DO NOT write on the question sheets.

- Write your name on the answer sheet.

- CIRCLE the letter(s) of your answer choice(s) next to the corresponding question number ON THE ANSWER SHEET.

- Some items have more than one correct choice.

- Wrong answers will be subtracted from correct answers to arrive at a final score for each item.

* * * * * * * * * * * * * * *

1. A "representative" water sample represents

   a. the best water quality area of the source at the time of sampling.
   b. the conditions existing in the water quality of the source at the time of sampling.
   c. The worst water quality area of the source at the time of sampling.

2. Written procedures to provide an accurate record for the chain-of-custody of monitoring samples should be developed for

   a. sample collection and transport.
   b. sample collection, transport and delivery to a laboratory.
   c. sample collection, transport, delivery to a laboratory and distribution to analyst(s).
   d. sample collection, transport, delivery to a laboratory, distribution to analyst(s) and storage or disposal.

3. For NPDES samples, the first source of information about preservation measures to be applied are

   a. those developed by the responsible laboratory.
   b. those published in the source of the analytical method to be used.
   c. those published in the EPA "Methods for Chemical Analysis of Water and Wastes."
   d. those published in the Federal Register.

4. The first consideration when drawing up a time schedule for analyzing samples is

   a. the preservative used.
   b. the number of analysts available.
   c. the holding time guideline for the constituent of interest.
   d. the quality control checks to be used.
   e. the number of samples to be processed.

Some items have more than one correct choice.

5. Which of the following have the right to apply for permission to use a procedure different from those published in the Federal Register for NPDES or drinking water compliance analyses?
   - a. A permit holder
   - b. A representative of a monitoring agency
   - c. A representative of a certified laboratory
   - d. A representative of a non-certified laboratory
   - e. A representative of an instrument manufacturer

6. According to Federal Register regulations, which of the following apply/applies to field kits for NPDES compliance monitoring?
   - a. Field kits may never be used.
   - b. If a kit contains all the reagents and equipment listed in a referenced method, it may be used.
   - c. Field kits with non-referenced reagents and/or equipment are considered an alternative procedure and require specific permission for use.

7. Which of the following is/are precision statistics?
   - a. % relative error
   - b. % relative standard deviation
   - c. % bias
   - d. % recovery
   - e. range
   - f. standard deviation

8. Which of the following is/are related to control of accuracy?
   - a. Analysis of reagent blanks
   - b. Analysis of duplicate samples
   - c. Analysis of spiked samples
   - d. Analysis of standards to check reproducibility of a curve
   - e. Analysis using standard addition techniques

9. When an analyst begins a titration, which of the following is/are unknown?
   - a. The concentration in the sample
   - b. The concentration in the titrant (standard solution)
   - c. The volume of sample
   - d. The volume of titrant required

10. What is another name for a substance which is a proton acceptor?
    - a. acid
    - b. base
    - c. salt
    - d. isomer
    - e. polymer

Some items have more than one correct choice.

3

11. If a volume requirement is stated as "50.0" ml, which one of the following should be used to measure and deliver it?

    a. A graduated 50 ml beaker
    b. A graduated 50 ml erlenmeyer flask
    c. A 50 ml graduated cylinder
    d. A 50 ml volumetric pipet

12. In reference to volumetric analyses, which of the following is/are true?

    a. Volumetric analysis requires a completed chemical reaction.
    b. Volumetric analysis requires an indicator of when the correct volume of titrant has been used.
    c. Calculations for volumetric analyses represent the molarity relationships of the solutions involved in a chemical reaction.
    d. Volumetric analysis cannot be used to determine the weight of a constituent in a volume of sample.

13. The end point for an alkalinity determination on an NPDES sample is

    a. the neutralization product pH for organic acids.
    b. the neutralization product pH for mineral acids.
    c. the neutralization product pH for alkali metal ions.
    d. the neutralization product pH for hydroxide ions.
    e. the neutralization product pH for bicarbonate ions.

14. Which of the following describe(s) every buffer?

    a. It keeps the pH at 7.0
    b. It resists changes in pH.
    c. It controls volatile interferences.
    d. It keeps an acid pH.
    e. It keeps a basic pH.

15. Reaction completion in a total hardness titration produces a

    a. blue color.
    b. green color.
    c. red color.
    d. pH 4.5
    e. pH 8.2
    f. change in amperage.

16. What is the titrant (in the buret) for a total hardness determination?

    a. EDTA
    b. Combined reagent
    c. 0.02 N hydrochloric acid
    d. Phenylarsine oxide
    e. SPADNS reagent

Some items have more than one correct choice.

4

17. One indicator which is approved for use in a total hardness titration is

    a. phenolphthalein.
    b. starch.
    c. methyl red.
    d. Eriochrome Black-T (EBT).
    e. Formazin.

18. The end point in one of the total residual chlorine titration tests produces a

    a. blue-green color.
    b. green color.
    c. red color.
    d. pH 4.5
    e. pH 8.2
    f. change in amperage.

19. A back-titration must be used to accurately determine total residual chlorine in

    a. samples held beyond 6 hours.
    b. samples preserved with inorganic acids.
    c. samples requiring pH adjustments.
    d. samples with dissolved inorganic materials.
    e. samples with dissolved organic materials.

20. The recognized method(s) to determine total residual chlorine for NPDES purposes is/are:

    a. Back-titration with phenylarsine oxide titrant
    b. Back-titration with iodine titrant
    c. Direct titration with ferrous ammonium sulfate titrant
    d. Color comparison using a DPD kit

21. The analytical reaction used to determine total phosphorus produces a

    a. blue color.
    b. green color.
    c. red color.
    d. pH 4.5
    e. pH 8.2
    f. change in amperage.

22. The total phosphorus determination includes

    a. sulfuric acid hydrolysis.
    b. combined reagent addition.
    c. persulfate oxidation.
    d. pH control.
    e. developing a standard curve.

Some items have more than one correct choice.

23. To determine total phosphorus for NPDES requirements, all phosphorus in the sample must either already be in, or be converted to, which of the following forms?

    a. P
    b. P_2O_5
    c. H_3PO_4
    d. PO_4^{-3}

24. The analytical reaction used in the SPADNS determination of fluoride involves a

    a. blue color.
    b. green color.
    c. red color.
    d. pH 4.5
    e. pH 8.2
    f. change in amperage.

25. The distillation procedure for fluoride

    a. distills over interferences leaving F^- behind
    b. distills over F^- leaving interferences behind
    c. requires temperature measurements
    d. produces the color used for subsequent measurement
    e. requires fresh sulfuric acid for each sample

26. The fluoride electrode can be used with

    a. an expanded scale pH meter
    b. any pH meter
    c. a conductivity meter
    d. a specific ion meter
    e. a spectrophotometer with adapted connectors

27. The analytical reaction used in the cadmium reduction determination of nitrate and nitrite produces a

    a. blue color.
    b. green color.
    c. red color.
    d. pH 4.5
    e. pH 8.2
    f. change in amperage.

28. Interferences in the cadmium reduction method include

    a. temperature changes.
    b. grease clogging of the column.
    c. suspended solids in the column.
    d. the short-term usability of the cadmium.
    e. nitrate color interferences.

Some items have more than one correct choice.

6

29. In the cadmium reduction method, the analysis depends on a test for:

   a. ammonium ions.
   b. copper ions.
   c. cadmium ions.
   d. nitrate ions.
   e. nitrite ions.

30. For NPDES purposes, which of the following filters is/are approved for the determination of non-filterable (suspended) and filterable (dissolved) residues?

   a. asbestos
   b. paper fibers
   c. glass fibers
   d. membrane
   e. paper mat

31. The drying temperature specified for NPDES determinations of total and non-filterable (suspended) residues is

   a. 103-105°C
   b. 180°C
   c. 550°C

32. The NPDES methods for residue (solids) determinations would be classed as

   a. automated.
   b. gravimetric analyses.
   c. meter determinations.
   d. photometric colorimetry.

33. Before the final weight is calculated for suspended solids,

   a. only one weighing must be carried out after heating.
   b. only two weighings must be carried out after heating.
   c. a constant weight must be obtained after heating.
   d. agreement on results must be obtained with a turbidimeter.
   e. agreement on results must be obtained with a conductivity meter.

34. For NPDES or drinking water, turbidity is measured with

   a. an instrument containing a Wheatstone bridge.
   b. a reference solution to determine potential differences.
   c. a colorimeter.
   d. a nephelometer.
   e. a spectrophotometer.

35. Turbidity is a parameter of water quality interest because

    a. results are an accurate estimate of mg/L suspended solids.
    b. results are an accurate estimate of mg/L dissolved solids.
    c. results indicate possibility of interference for chlorine disinfection process.
    d. results are an index to the aesthetic quality of water.
    e. results indicate the efficiency of treatment processes.

36. Standardization of the instrument used to measure turbidity for NPDES or drinking water data involves the use of

    a. Formazin.
    b. Fuller's earth.
    c. a curve supplied by the manufacturer.
    d. a Jackson candle turbidimeter.
    e. Kaolin.

37. For NPDES, specific conductivity is measured with

    a. an instrument containing a Wheatstone bridge.
    b. a reference solution to determine potential differences.
    c. a colorimeter.
    d. a nephelometer.
    e. a spectrophotometer.

38. Specific conductivity is a parameter of water quality interest because

    a. results are an estimate of mg/L suspended solids.
    b. results are an estimate of mg/L dissolved solids.
    c. results are used to determine flow patterns.
    d. results are used to signal "dumps" into a plant influent.
    e. results are used to check the purity of distilled and deionized water.

39. Which of the following affect(s) the results for specific conductivity measurements?

    a. the cell constant
    b. the color of the sample
    c. the temperature
    d. the number and kind of ions in a sample
    e. the resistivity of sample components

40. ON THE ANSWER SHEET, list four titles (or the publishers) of books of chemical procedures which are recognized in the Federal Register as sources of nationally-tested analytical methods applicable to water samples.

INORGANIC ANALYSES IN WATER QUALITY PROGRAMS (100.4)

ANSWER SHEET

NAME ______________________________ DATE __________________

1. a b c
2. a b c d
3. a b c d
4. a b c d e
5. a b c d e

6. a b c
7. a b c d e f
8. a b c d e
9. a b c d
10. a b c d e

11. a b c d
12. a b c d
13. a b c d e
14. a b c d e
15. a b c d e f

16. a b c d e
17. a b c d e
18. a b c d e f
19. a b c d e
20. a b c d

21. a b c d e f
22. a b c d e
23. a b c d
24. a b c d e f
25. a b c d e

26. a b c d e

27. a b c d e f

28. a b c d e

29. a b c d e

30. a b c d e

31. a b c

32. a b c d

33. a b c d e

34. a b c d e

35. a b c d e

36. a b c d e

37. a b c d e

38. a b c d e

39. a b c d e

40. a.

b.

c.

d.

INORGANIC ANALYSIS IN WATER QUALITY PROGRAMS
EVALUATION

SCORING INFORMATION

There are 40 items on the test. Each item was scored as an entity and had a maximum value of 2½ points. Within each item, any wrong* answers were subtracted from the correct answers, and the remainder (zero was the minimum used) was given the following credit for the item:

1. For items requiring only one correct answer:
   a. a remainder of 1, 2½ points
2. For items requiring two correct answers:
   a. a remainder of 1, 1¼ points
3. For items requiring three correct answers:
   a. a remainder of 2 or 3, 2½ points
   b. a remainder of 1, 1¼ points
4. For items requiring four correct answers:
   a. a remainder of 3 or 4, 2½ points
   b. a remainder of 2, 1¼ points
   c. a remainder of 1, no credit
5. For items requiring five correct answers:
   a. a remainder of 4 or 5, 2½ points
   b. a remainder of 2 or 3, 1¼ points
   c. a remainder of 1, no credit

*NOTE: Missing answers were not specifically counted. The scoring system is based on the total number of answers that should be given and thereby accounts for any missing answers.

KEY

INORGANIC ANALYSES IN WATER QUALITY PROGRAMS (100.4)
ANSWER SHEET

NAME ______________________________ DATE ______________________

1. a [✓ b] c
2. a b c [✓ d] (Must have d circled. If d plus others are circled, that's OK).
3. a b c d (11/80, b is answer. Check if proposed d has been finalized).
4. a b [✓ c] d e
5. [✓ a] [✓ b] [✓ c] [✓ d] [✓ e]
6. a [✓ b] [✓ c]
7. a [✓ b] c d [✓ e] [✓ f]
8. [✓ a] b [✓ c] d [✓ e]
9. [✓ a] b c [✓ d]
10. a [✓ b] c d e
11. a b c [✓ d]
12. [✓ a] [✓ b] c d
13. a b c d [✓ e]
14. a [✓ b] c d e
15. [✓ a] b c d e f
16. [✓ a] b c d e
17. a b c [✓ d] e
18. a b c d e [✓ f]
19. a b c d [✓ e]
20. a [✓ b] [✓ c] d
21. [✓ a] b c d e f
22. [✓ a] [✓ b] [✓ c] [✓ d] [✓ e]
23. a b c [✓ d]
24. a b [✓ c] d e f
25. a [✓ b] [✓ c] d e

26. [✓ a] b c [✓ d] e

27. a b [✓ c] d e f

28. a [✓ b] [✓ c] d e

29. a b c d [✓ e]

30. a b [✓ c] d e

31. [✓ a] b c

32. a [✓ b] c d

33. a b [✓ c] d e

34. a b c [✓ d] e

35. a b [✓ c] [✓ d] [✓ e]

36. [✓ a] b c d e

37. [✓ a] b c d e

38. a [✓ b] [✓ c] [✓ d] [✓ e]

39. [✓ a] b [✓ c] [✓ d] [✓ e]

40. a. Methods for Chemical Analysis of Water and Wastes
or EPA

b. Standard Methods for the Examination of Water and Wastewater
or Standard Methods
or APHA-AWWA-WPCF

c. Annual Book of ASTM Standards (Part 31)
or ASTM

d. U.S. Geological Survey Techniques of Water-Resources Investigations
or Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases
or USGS

Date

Name
Address

Dear

Our current policy is not to award continuing education units for the course, Inorganic Analyses in Water Quality Control Programs, January 9-13, 1978 unless a score of 50 or above on the pre-course evaluation is increased by 20 points (or more to a maximum required final score of 90) on the post-course evaluation. Your scores showed an increase of 17 points, so the units are not recorded on your certificate.

The course evaluation is our objective basis for awarding continuing education units for the course. However, we realize that each person who attends has an individual background of interests and individual goals to be achieved. The evaluation items do not necessarily measure the accomplishment of these individual goals nor all the improvements of knowledge and skills derived by each person in the class.

Your effort and interest in all the course activities indicated that you must have been successful in achieving your personal goals. It was really a pleasure to have you in the class and we hope you will let us know if we can be of further service to you.

Sincerely yours,

Name
Course Coordinator
National Training and Operational
Technology Center

Enclosure - Certificate

Date

Name
Address

Dear

Our current policy is not to award continuing education units or course certificates to those who do not attend at least 95% of the lecture/laboratory sessions.

This policy will apply to you, since your illness prevented your participation in the Tuesday and Wednesday activities of the course, Inorganic Analyses in Water Quality Control Programs, January 9-13, 1978.

I would, however, like to acknowledge your attendance and full participation in the other three days of the course. It was really a pleasure to have you in the class and we hope you will let us know if we can be of further service to you.

Sincerely yours,

Name
Course Coordinator
National Training and Operational
Technology Center

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Sample Handling - Field Through Laboratory

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: A basic element of quality assurance for chemical analytical data is a reliable and valid sample, properly preserved, stored and documented. An analyst must be knowledgeable about sample handling to carry out his/her responsibilities.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of inorganic chemistry

B. Though not essential, it would aid comprehension/retention of information if the participant has some experience in chemical laboratory work involving water samples.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will have an overview of all the elements of a program to assure a valid, properly preserved sample, whose integrity is documented from collection to storage.

B. Conditions: He/she will be given the training manual, handouts (VII. A. 4. and 5.) regarding sample preservation techniques, and a 30 minute discussion of the elements involved.

C. Accepted Performance: He/she must participate in the discussion and correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the training manual, "Sample Handling - Field Through Laboratory".

2. (Optional) Copies for display of the standard reference books of methods listed in the training manual outline section, "VI Methods of Analysis".

3. Eight slides, X-17: Sample Handling, listing items from divisions of the outline in the training manual. (See XI Description of Visual Materials).

4. Handout: The preservation procedures for samples collected for National Pollutant Discharge Elmination System or State

Certification report purposes are to be finalized in the Federal Register sometime after January, 1981. To ensure that you distribute current information, contact your EPA Regional Quality Assurance Coordinator for copies of the appropriate information.

5. Handout: The preservation procedures specifically designed for drinking water samples have been published in the U.S. EPA Report No. EPA 600/8-78-008, May, 1978. A copy of the applicable information is at the end of this IPW.

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review visual materials and available handouts.

2. Prepare the lesson using the Sequencing below or your own organization of the material.

3. Duplicate copies of any handout materials.

B. Sequencing:

1. Follow the outline in the training manual for discussion. Participants should use the outline, too. The slides, X-17, Sample Handling, can be used to guide the discussion according to the divisions in the training manual outline. The divisions are referenced in the following:

a. I Planning a Sample Program

1) A. Discuss the factors to consider - slide 1
Define grab and composite samples - slide 2

B. Decisive Criteria for making decisions should be woven into the discussion.

b. II Representative Samples - Associated factors - slide 3

c. III Sample Identification - Topics for discussion - slide 4

d. IV Containers - Considerations for discussion - slide 5

e. V Preservation, A. Functions - slide 6
B. General Methods - slide 7
C. Specific Methods - Note sources of information
D. Federal Register Methods - Distribute handouts and discuss.

f. VI Names of Standard References for Methods - Display a copy as each title is given.
   E. Federal Register Methodology - Note that there are regulations for NPDES, State Certifications and Drinking Water. This topic is covered in detail in a separate lesson, Compliance Methodology.

g. VII Order of Analyses - Based on holding times which depend on factors listed on slide 8.
   A. Give examples of different holding times
      Give sources of information

h. VIII Record Keeping
   A. Field - kind of information
   B. Lab - kind of information

i. IX Summary (as in outline)

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For each student:

1. One copy of handout material

X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Eight slides, X-17: Sample Handling - See next page.

SERIES X-17: SAMPLE HANDLING

COLLECTION OF SAMPLES

- SAMPLING SITE
- EQUIPMENT
- QUANTITY
- TYPE
- PRESERVATION

SLIDE 1

TYPES OF SAMPLES

GRAB SAMPLE - INDICATES CONDITION AT TIME OF SAMPLING

COMPOSITE SAMPLE - INDICATES AVERAGE CONDITION OVER COMPOSITING PERIOD

SLIDE 2

REPRESENTATIVE SAMPLES

- CLEAN CONTAINER
- COMPOSITING SAMPLES
- PROPER EQUIPMENT
- SLUDGE OR MUD

SLIDE 3

SAMPLE IDENTIFICATION

- PROPER TAG
- USEFUL IDENTIFICATION INFORMATION
- SAMPLE CUSTODY

SLIDE 4

SAMPLE CONTAINERS

- AVAILABLE MATERIALS
- CHEMICAL AND PHYSICAL INTERACTIONS
- MAILING REQUIREMENTS PRELIMINARY CHECK
- CLEANING
- STORAGE

SLIDE 5

SAMPLE PRESERVATION

- RETARD BIOLOGICAL ACTION
- RETARD PRECIPITATION OR HYDROLYSIS
- REDUCE VOLATILITY

SLIDE 6

GENERAL METHODS OF SAMPLE PRESERVATION

- pH CONTROL
- CHEMICAL ADDITION
- REFRIGERATION OR FREEZING

SLIDE 7

TIME INTERVAL BETWEEN COLLECTION AND ANALYSIS

DEPENDS ON -SAMPLE TYPE
- STABILITY
- STORAGE CONDITIONS

SLIDE 8

SLIDE ______

From: EPA 600/8-78-008, May 1978 "Manual for Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies"

Table IV-2.—*Sample collecting, handling, and preservation*[1]

Parameter	Preservative[2]	Container[3]	Maximum holding time[4]
Arsenic	Conc HNO_3 to $pH<2$	P or G	6 months
Barium	Conc HNO_3 to $pH<2$	P or G	6 months
Cadmium	Conc HNO_3 to $pH<2$	P or G	6 months
Chromium	Conc HNO_3 to $pH<2$	P or G	6 months
Lead	Conc HNO_3 to $pH<2$	P or G	6 months
Mercury	Conc HNO_3 to $pH<2$	G P	38 days 14 days
Nitrate	Conc H_2SO_4 to $pH<2$	P or G	14 days
Selenium	Conc HNO_3 to $pH<2$	P or G	6 months
Silver	Conc HNO_3 to $pH<2$	P or G	6 months
Fluoride	None	P or G	1 month
Chlorinated hydrocarbons	Refrigerate at 4° C as soon as possible after collection	G with foil- or Teflon-lined cap	14 days[5]
Chlorophenoxys	Refrigerate at 4° C as soon as possible after collection	G with foil- or Teflon-lined cap	7 days[5]

[1] If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and so notify the authority requesting the analyses.

[2] If HNO_3 cannot be used because of shipping restrictions, sample may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with conc HNO_3 to $pH<2$. At time of analysis, sample container should be thoroughly rinsed with 1:1 HNO_3; washings should be added to sample.

[3] P = Plastic, hard or soft; G = Glass, hard or soft.

[4] In all cases, samples should be analyzed as soon after collection as possible.

[5] Well-stoppered and refrigerated extracts can be held up to 30 days.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Compliance Methodology

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 70 minutes (60 + 10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: A basic element of quality assurance for chemical analytical data is use of a recognized analytical procedure. In addition, federal regulations affect the final choice of methodology for compliance samples. An analyst must know about these regulations to fulfill the attendant responsibilities.

V. ENTRY LEVEL BEHAVIOR:

A. Familiarity with fundamentals of chemical analytical methods

B. Though not essential, it is orderly to have completed the lesson (or equivalent):

1. Sample Handling

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will be able to use Federal Registers and pertinent EPA explanatory memos to choose approved analytical methodology for compliance with the National Pollutant Discharge Elimination System and the National Interim Primary Drinking Water Regulations.

B. Conditions: He/she will be given the training manual, copies of the pertinent information (See VII A), a 60 minute explanation of their applicability and format and 10 minutes at a later time to resolve any questions.

C. Accepted Performance: Satisfactory according to an Instructor rating of attention and use of the materials as directed during the discussion. He/she must also correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the training manual:

a. "Methodology for Chemical Analysis of Water and Wastewater" is a glossary of the methods promulgated for obtaining compliance data.

2. Handouts:

   a. National Pollutant Discharge Elimination System - NPDES:

      1) Guidelines Establishing Test Procedures, 40 CFR 136, Federal Register, 12/1/76, pp 52780-52786. A copy is at the end of this IPW, and also an errata sheet. Changes were proposed 12/18/79, pp. 75028-75052 and should be finalized after January, 1981. Check with your EPA Regional Quality Assurance Coordinator to see if the finalized Guidelines have been published. (Name and number is available from EMSL-Cincinnnati, 513/684-7301). The Coordinator can supply copies to you.

      2) Summary of protocols to apply for approval of an alternative test procedure for regulatory purposes. A copy is at the end of this IPW.

      3) Federal Register notices of alternative methods approved to date (8/80) are at the end of this IPW. Contact your EPA Regional Quality Assurance Coordinator for copies of notices published since then.

      4) EPA-EMSL memo on "Use of Chemical Test Kits for Compliance Monitoring." A copy is at the end of this IPW.

      5) EPA-EMSL memo on "Use of "Prepared" Reagents in NPDES Compliance." A copy is at the end of this IPW.

   b. National Interim Primary Drinking Water Regulations - NIPDWR:

      1) NIPDWR, 40 CFR 141, Federal Register, 12/24/75, pp 59566-59574. A copy is at the end of this IPW.

      2) Amendments to NIPDWR were proposed 7/19/79, 40 CFR 141. The amendments were finalized 40 CFR Part 141, Federal Register, 8/27/80, pp. 57332-57346. Copies of pp. 57344-57346 are at the end of this IPW. The pages contain the current (8/80), approved analytical requirements for drinking water analysis. These include an update of the approved methods presented in the 12/24/75 NIPDWR and also alternate analytical techniques approved by EPA since that date. Check with your EPA Regional Quality Assurance Coordiantor to get copies of any additional, pertinent Federal Register notices published after 8/80.

   c. National Secondary Drinking Water Regulations - NSDWR:

      1) NSDWR, 40 CFR 143, Federal Register, pp 42198-42199. A copy is at the end of this IPW.

B. Suggested Media:

1. OPTIONAL: Copies of the methods manuals cited for NPDES and NIPDWR could be available for display/inspection.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Contact your EPA Regional Quality Assurance Coordinator to check if any Federal Registers pertaining to NPDES or NIPDWR have been issued since the ones referenced in VII. A. above (8/80). If so, obtain copies from the Coordinator for your class.

2. Thoroughly review all pertinent Federal Register information and handout materials. Since the regulations are legally binding, it is imperative to impart correct and current information to the participants.

3. Prepare the lesson using the Sequencing below or your own organization of the materials.

4. Duplicate copies of all the handout materials. Most can be copied back-to-back, but be careful to keep each handout as an entity so participants can use each independent of the other. Collate the handouts into a packet for each participant to minimize distribution time.

B. Sequencing:

1. Hand out the packets of information.

2. Introduction

   a. Choice of methodology affects validity and reliablity of data.

   b. Analytical programs affected by federal legislation include those involving point source discharges (NPDES) and drinking water (NIPDWR).

3. National Pollutant Discharge Elimimation System (NPDES). CAUTION: Use the current Federal Register. The NPDES Guidelines are scheduled for an update after January, 1981.

   a. Applicability of the regulations. See 12/1/76, page 52780, paragraph 2.

   b. Overview of Table 1

      1) Column 1: Alphabetical listing of parameters and units. Note subcategories.

      2) Column 2: Method listings and possibly specification of a pretreatment and/or method choice, e.g. acidity end point is to be pH of 8.2. A summary of the principles involved in the listed methodology can be found in the training manual outline, "Methodology for Chemical Analysis of Water and Wastewater." You can use the outline to list the types of methodology found in the Table. Note that many automated methods are approved.

3) Remaining columns list sources (with page numbers) of approved methods. You can display copies of the cited manuals at this time and leave them to be available for inspection by participants.

4) Note the location (by number) of the parameters to be taught during the course. Also note the method to be used for course laboratory sessions.

5) Note any errata (handout).

c. Go over highlights of Applying for Approval of Alternative Test Procedures (handout).

1) Limited use

2) Nationwide use

d. Go through the Federal Register notices of alternative methods approved for nationwide use, briefly noting the contents.

e. Go over highlights of the memo on "Use of Chemical Test Kits for Compliance Monitoring" (handout).

f. Note highlights of the memo on "Use of "Prepared" Reagents in NPDES Compliance" (handout).

4. Drinking Water Regulations. CAUTION: Use the current Federal Registers. The 12/24/75 Federal Register is the current (8/80) National Interim Primary Drinking Water Regulation (NIPDWR), along with the Amendments to NIPDWR which were finalized 8/27/80.

a. Applicability of the regulations. See 12/24/75, page 59566, "Water Systems Covered," and page 59570, "Subpart A - General."

b. Maximum Contaminant Levels are listed in 12/24/75 for Chemicals on page 59570 and for Turbidity on page 59571.

c. The analytical methodology in the 12/24/75 regulation was updated and expanded in the 8/27/80 regulation. Therefore, use pp. 57344-57346 from the 8/27/80 Federal Register to cite the location of the listings of approved methods: 141.22 Turbidity, 141.23 Inorganics, 141.24 Organics, 141.41 Sodium, 141.42 Corrosivity Characteristics. Also highlight section 141.28, Approved Laboratories, and section 141.27, Alternate Analytical Techniques. (Alternate methods approved after the original NIPDWR (12/24/75) are included in the sections just cited).

d. There are "National Secondary Drinking Water Regulations" which were finalized in the 7/19/79 Federal Register on pp. 42198-42199. These represent reasonable goals for the aesthetic quality of drinking water but are not to be federally enforceable. Suggested Maximum Contaminant Levels and Analytical Methodology are presented on these pages.

5. A 10 minute discussion period should be scheduled later to answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For each student:

1. Copies of handout materials

X. IPW REAGENT REQUIREMENTS:

A. None

federal register

WEDNESDAY, DECEMBER 1, 1976

THE NATIONAL ARCHIVES OF THE UNITED STATES 1934

PART II:

ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

Guidelines Establishing Test Procedures for the Analysis of Pollutants

Amendments

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

[FRL 630-4]

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Amendment of Regulations

On June 9, 1975, proposed amendments to the Guidelines Establishing Test Procedures for the Analysis of Pollutants (40 CFR 136) were published in the FEDERAL REGISTER (40 FR 24535) as required by section 304(g) of the Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92-500, 1972) hereinafter referred to as the Act.

Section 304(g) of the Act requires that the Administrator shall promulgate guidelines establishing test procedures for the analysis of pollutants that shall include factors which must be provided in: (1) any certification pursuant to section 401 of the Act, or (2) any permit application pursuant to section 402 of the Act. Such test procedures are to be used by permit applicants to demonstrate that effluent discharges meet applicable pollutant discharge limitations and by the States and other enforcement activities in routine or random monitoring of effluents to verify compliance with pollution control measures.

Interested persons were requested to submit written comments, suggestions, or objections to the proposed amendments by September 7, 1975. One hundred and thirty-five letters were received from commenters. The following categories of organizations were represented by the commenters: Federal agencies accounted for twenty-four responses; State agencies accounted for twenty-six responses; local agencies accounted for seventeen responses; regulated major dischargers accounted for forty-seven responses; trade and professional organizations accounted for eight responses; analytical instrument manufacturers and vendors accounted for seven responses; and analytical service laboratories accounted for six responses.

All comments were carefully evaluated by a technical review committee. Based upon the review of comments, the following principal changes to the proposed amendments were made:

(A) *Definitions.* Section 136.2 has been amended to update references: Twenty commenters, representing the entire spectrum of responding groups pointed out that the references cited in §§ 136.2(f), 136.2(g), and 136.2(h) were out-of-date; §§ 136.2(f), 136.2(g), and 136.2(h), respectively, have been amended to show the following editions of the standard references: "14th Edition of Standard Methods for the Examination of Water and Waste Water;" "1974 EPA Manual of Methods for the Analysis of Water and Waste;" and "Part 31, 1975 Annual Book of ASTM Standards."

(B) *Identification of Test Procedures.* Both the content and format of § 136.3, "Table I, List of Approved Test Procedures" have been revised in response to twenty-one comments received from State and local governments, major regulated dischargers, professional and trade associations, and analytical laboratories.

Table I has been revised by:

(1) The addition of a fourth column of references which includes procedures of the United States Geological Survey which are equivalent to previously approved methods.

(2) The addition of a fifth column of miscellaneous references to procedures which are equivalent to previously approved methods.

(3) Listing generically related parameters alphabetically within four subcategories: bacteria, metals, radiological and residue, and by listing these subcategory headings in alphabetic sequence relative to the remaining parameters.

(4) Deleting the parameter "Algicides" and by entering the single relevant algicide, "Pentachlorophenol" by its chemical name.

(C) *Clarification of Test Parameters.* The conditions for analysis of several parameters have been more specifically defined as a result of comments received by the Agency:

(1) In response to five commenters representing State or local governments, major dischargers, or analytical instrument manufacturers, the end-point for the alkalinity determination is specifically designated as pH 4.5.

(2) Manual digestion and distillation are still required as necessary preliminary steps for the Kjeldahl nitrogen procedure. Analysis after such distillation may be by Nessler color comparison, titration, electrode, or automated phenolate procedures.

(3) In response to eight commenters representative of Federal and State governments, major dischargers, and analytical instrument manufacturers, manual distillation at pH 9.5 is now specified for ammonia measurement.

(D) *New Parameters and Analytical Procedures.* Forty-four new parameters have been added to Table I. In addition to the designation of analytical procedures for these new parameters, the following modifications have been made in analytical procedures designated in response to comments.

(1) The ortho-tolidine procedure was not approved for the measurement of residual chlorine because of its poor accuracy and precision. Its approval had been requested by seven commenters representing major dischargers, State, or local governments, and analytical instrument manufacturers. Instead, the N,N-diethyl-p-phenylenediamine (DPD) method is approved as an interim procedure pending more intensive laboratory testing. It has many of the advantages of the ortho-tolidine procedure such as low cost, ease of operation, and also is of acceptable precision and accuracy.

(2) The Environmental Protection Agency concurred with the American Dye Manufacturers' request to approve its procedure for measurement of color, and copies of the procedure are now available at the Environmental Monitoring and Support Laboratory. Cincinnati (EMSL-CI).

(3) In response to three requests from Federal, State governments, and dischargers, "hardness," may be measured as the sum of calcium and magnesium analyzed by atomic absorption and expressed as their carbonates.

(4) The proposal to limit measurement of fecal coliform bacteria in the presence of chlorine to only the "Most Probable Number" (MPN) procedure has been withdrawn in response to requests from forty-five commenters including State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical supplies. The membrane filter (MF) procedure will continue to be an approved technique for the routine measurement of fecal colifiorm in the presence of chlorine. However, the MPN procedure must be used to resolve controversial situations. The technique selected by the analyst must be reported with the data.

(5) A total of fifteen objections, representing the entire spectrum of commenters, addressed the drying temperatures used for measurement of residues. The use of different temperatures in drying of total residue, dissolved residue and suspended residue was cited as not allowing direct intercomparability between these measurements. Because the intent of designating the three separate residue parameters is to measure separate waste characteristics (low drying temperatures to measure volatile substances, high drying temperatures to measure anhydrous inorganic substances), the difference in drying temperatures for these residue parameters must be preserved.

(E) *Deletion of Measurement Techniques.* Some measurement techniques that had been proposed have been deleted in response to objections raised during the public comment period.

(1) The proposed infrared spectrophotometric analysis for oil and grease has been withdrawn. Eleven commenters representing Federal or State agencies and major dischargers claimed that this parameter is defined by the measurement procedure. Any alteration in the procedure would change the definition of the parameter. The Environmental Protection Agency agreed.

(2) The proposed separate parameter for sulfide at concentrations below 1 mg/l, has been withdrawn. Methylene blue spectrophotometry is now included in Table I as an approved procedure for sulfide analysis. The titrimetric iodine procedure for sulfide analysis may only be used for analysis of sulfide at concentrations in excess of one milligram per liter.

(F) *Sample Preservation and Holding Times.* Criteria for sample preservation and sample holding times were requested by several commenters. The reference for sample preservation and holding time criteria applicable to the Table I parameters is given in footnote (1) of Table I.

(G) *Alternate Test Procedures.* Comments pertaining to § 136.4, Application for Alternate Test Procedures, included objections to various obstacles within these procedures for expeditious approval of alternate test procedures. Four analytical instrument manufacturers commented that by limiting of application for review and/or approval of alternate test procedures to NPDES permit holders, § 136.4 became an impediment to the commercial development of new or improved measurement devices based on new measurement principles. Applications for such review and/or approval will now be accepted from any person. The intent of the alternate test procedure is to allow the use of measurement systems which are known to be equivalent to the approved test procedures in waste water discharges.

Applications for approval of alternate test procedures applicable to specific discharges will continue to be made only by NPDES permit holders, and approval of such applications will be made on a case-by-case basis by the Regional Administrator in whose Region the discharge is made.

Applications for approval of alternate test procedures which are intended for nationwide use can now be submitted by any person directly to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati. Such applications should include a complete methods write-up, any literature references, comparability data between the proposed alternate test procedure and those already approved by the Administrator. The application should include precision and accuracy data of the proposed alternate test procedure and data confirming the general applicability of the test procedure to the industrial categories of waste water for which it is intended. The Director of the Environmental Monitoring and Support Laboratory, after review of submitted information, will recommend approval or rejection of the application to the Administrator, or he will return the application to the applicant for more information. Approval or rejection of applications for test procedures intended for nationwide use will be made by the Administrator, after considering the recommendation made by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. Since the Agency considers these procedures for approval of alternate test procedures for nationwide use to be interim procedures, we will welcome suggestions for criteria for approval of alternate test procedures for nationwide use. Interested persons should submit their written comments in triplicate on or before June 1, 1977 to: Dr. Robert B. Medz, Environmental Protection Technologist, Monitoring Quality Assurance Standardization, Office of Monitoring and Technical Support (RD-680), Environmental Protection Agency, Washington, D.C. 20460.

(H) *Freedom of Information.* A copy of all public comments, an analysis by parameter of those comments, and documents providing further information on the rationale for the changes made in the final regulation are available for inspection and copying at the Environmental Protection Agency Public Information Reference Unit, Room 2922, Waterside Mall, 401 M Street, SW., Washington, D.C. 20460, during normal business hours. The EPA information regulation 40 CFR 2 provides that a reasonable fee may be charged for copying such documents.

Effective date: These amendments become effective on April 1, 1977.

Dated: November 19, 1976.

JOHN QUARLES,
Acting Administrator,
Environmental Protection Agency.

Chapter I, Subchapter D, of Title 40, Code of Federal Regulations is amended as follows:

1. In § 136.2, paragraphs (f), (g), and (h) are amended to read as follows:

§ 136.2 Definitions.

* * * * *

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water,* 14th Edition, 1976. This publication is available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 31, Water, 1975.* This publication is available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Waste, 1974.* Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, Cincinnati, Ohio 45268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

2. In § 136.3, the second sentence of paragraph (b) is amended, and a new paragraph (c) is added to read as follows:

§ 136.3 Identification of test procedures.

* * * * *

(b) * * * Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or the Director upon the recommendation of the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring and Support Laboratory, Cincinnati, additional alternate test procedures for nationwide use.

3. Table I of § 136.3 is revised by listing the parameters alphabetically; by adding 44 new parameters; by adding a fourth column under references listing equivalent United States Geological Survey methods; by adding a fifth column under references listing miscellaneous equivalent methods; by deleting footnotes 1 through 7 and adding 24 new footnotes, to read as follows:

* * * * *

TABLE I.—*List of approved test procedures*[1]

Parameter and units	Method	1974 EPA methods	References (page nos.) 14th ed. standard methods	Pt. 31 1975 ASTM	USGS methods[2]	Other approved methods
1. Acidity, as $CaCO_3$, milligrams per liter.	Electrometric end point (pH of 8.2) or phenolphthalein end point.	1	273(4d)	116	40	[3](607)
2. Alkalinity, as $CaCO_3$, milligrams per liter.	Electrometric titration (only to pH 4.5) manual or automated, or equivalent automated methods.	3 5	278	111	41	[3](607)
3. Ammonia (as N), milligrams per liter.	Manual distillation[4] (at pH 9.5) followed by nesslerization, titration, electrode, Automated phenolate.	159 165 168	410 412 616	237	116	[3](614)
BACTERIA						
4. Coliform (fecal)[5], number per 100 ml.	MPN;[6] membrane filter		922			
5. Coliform (fecal)[5] in presence of chlorine, number per 100 ml.	do.[6] [7]		937 922 928, 937		[7](45)	
6. Coliform (total),[5] number per 100 ml.	do.[6]		916			
7. Coliform (total)[5] in presence of chlorine, number per 100 ml.	MPN;[6] membrane filter with enrichment.		928 916 933		[7](35)	
8. Fecal streptococci,[5] number per 100 ml.	MPN;[6] membrane filter; plate count.		943 944 947		[7](50)	
9. Benzidine, milligrams per liter.	Oxidation—colorimetric[8]					
10. Biochemical oxygen demand, 5-d (BOD_5), milligrams per liter.	Winkler (Azide modification) or electrode method.		543		[7](50)	
11. Bromide, milligrams per liter	Titrimetric, iodine-iodate	14		323	58	[10](17)
12. Chemical oxygen demand (COD), milligrams per liter.	Dichromate reflux	20	550	472	124	[9](610)
13. Chloride, milligrams per liter	Silver nitrate; mercuric nitrate; or automated colorimetric-ferricyanide.	29 31	303 304 613	267 265	[11](46)	[10](17) [9](615)

See footnotes at end of table.

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods [3]	
14. Chlorinated organic compounds (except pesticides), milligrams per liter.	Gas chromatography [13]					
15. Chlorine—total residual, milligrams per liter.	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or Titrimetric methods (these last 2 are interim methods pending laboratory testing).	35	318 322 332 329	278		
16. Color, platinum cobalt units or dominant wave length, hue, luminance, purity.	Colorimetric; spectrophotometric; or ADMI procedure.[12]	36 39	64 66		82	
17. Cyanide, total,[14] milligrams per liter.	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric.	40	361	503	85	[10](22)
18. Cyanide amenable to chlorination, milligrams per liter.	do	49	376	505		
19. Dissolved oxygen, milligrams per liter.	Winkler (Azide modification) or electrode method.	51 56	443 450	368	126	[2](600)
20. Fluoride, milligrams per liter.	Distillation [4] followed by ion electrode; SPADNS; or automated complexone.	65 59 61	389 391 393 614	307 305	93	
21. Hardness—Total, as $CaCO_3$, milligrams per liter.	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates).	68 70	202	161	94	[2](617)
22. Hydrogen ion (pH), pH units.	Electrometric measurement.	239	460	178	129	[2](606)
23. Kjeldahl nitrogen (as N), milligrams per liter.	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate.	175 165 182	437		122	[2](612)
METALS						
24. Aluminum—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Eriochrome Cyanine R).	92	152 171		[10] (19)	
25. Aluminum—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced methods for total aluminum.					
26. Antimony—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	94				
27. Antimony—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total antimony.					
28. Arsenic—Total, milligrams per liter.	Digestion followed by silver diethyldithiocarbamate; or atomic absorption.[16] [18]	9 95	285 283 159		[10] (31) [10] (37)	
29. Arsenic—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total arsenic.					
30. Barium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	97	152		52	
31. Barium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total barium.					
32. Beryllium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Aluminon).	99	152 177		58	
33. Beryllium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total beryllium.					
34. Boron—Total, milligrams per liter.	Colorimetric (Curcumin)	13	287			
35. Boron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total boron.					
36. Cadmium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	101	148 182	345	62	[3] (619) [10] (37)
37. Cadmium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cadmium.					
38. Calcium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption; or EDTA titration.	103	148 189	345	66	
39. Calcium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total calcium.					
40. Chromium VI, milligrams per liter.	Extraction and atomic absorption; colorimetric (Diphenylcarbazide).	89, 105	192		76 75	
41. Chromium VI—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for chromium VI.					
42. Chromium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Diphenylcarbazide).	105	148 192	345 286	78 77	[3] (619)
43. Chromium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total chromium.					

See footnotes at end of table.

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods [3]	
44. Cobalt—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	107	148	345	80	[19] (37)
45. Cobalt—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cobalt.					
46. Copper—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Neocuproine).	108	148 196	345 203	83	[5] (619) [19] (37)
47. Copper-Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total copper.					
48. Gold—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
49. Iridium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
50. Iron—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Phenanthroline).	110	148 208	345 326	102	[5] (619)
51. Iron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total iron.					
52. Lead—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	112	148 215	345	105	[5] (619)
53. Lead—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total lead.					
54. Magnesium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption; or gravimetric.	114	148 221	345	109	[5] (619)
55. Magnesium—Dissolved milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total magnesium.					
56. Manganese—Total milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Persulfate or periodate).	116	148 225, 227	345	111	[5] (619)
57. Manganese—Dissolved milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total manganese.					
58. Mercury—Total, milligrams per liter.	Flameless atomic absorption.	118	156	386	[20] (51)	
59. Mercury—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total mercury.					
60. Molybdenum—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	139		350		
61. Molybdenum—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total molybdenum.					
62. Nickel—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Heptoxime).	141	148	345	115	
63. Nickel—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total nickel.					
64. Osmium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
65. Palladium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
66. Platinum—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
67. Potassium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption, colorimetric (Cobaltinitrite), or by flame photometric.	143	235 234	406	134	[5] (620)
68. Potassium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced methof for total potassium.					
69. Rhodium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
70. Ruthenium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]					
71. Selenium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[18] [19]	145	159			
72. Selenium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total selenium.					
73. Silica—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by colorimetric (Molybdosilicate).	274	487	398	139	
74. Silver—Total,[20] milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	146	148 243		142	[5](619) [19](37)
75. Silver—Dissolved,[20] milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total silver.					
76. Sodium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption or by flame photometric.	147	250	403	148	[5] (621)
77. Sodium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total sodium.					

See footnotes at end of table

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.) Pt. 31 1975 ASTM	References (page nos.) USGS methods [2]	Other approved methods
78. Thallium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	149				
79. Thallium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total thallium.					
80. Tin—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	150			[11] (65)	
81. Tin—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total tin.					
82. Titanium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	151				
83. Titanium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total titanium.					
84. Vanadium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Gallic acid).	153	152 260	441	[11] (67)	
85. Vanadium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total vanadium.					
86. Zinc—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	155	148 265	345	159	[3](619) [19](37)
87. Zinc—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total zinc.					
88. Nitrate (as N), milligrams per liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.[21]	201 197 207	423 427 620	358	119	[3](614) [19](28)
89. Nitrite (as N), milligrams per liter.	Manual or automated colorimetric (Diazotization).	215	434		121	
90. Oil and grease, milligrams per liter.	Liquid-liquid extraction with trichloro-trifluoroethane-gravimetric.	229	515			
91. Organic carbon; total (TOC), milligrams per liter.	Combustion—Infrared method.[22]	236	532	467	[20] (4)	
92. Organic nitrogen (as N), milligrams per liter.	Kjeldahl nitrogen minus ammonia nitrogen.	175, 159	437		122	[3] (612, 614)
93. Orthophosphate (as P), milligrams per liter.	Manual or automated ascorbic acid reduction.	249 256	481 624	384	131	[3] (621)
94. Pentachlorophenol, milligrams per liter.	Gas chromatography [12]					
95. Pesticides, milligrams per liter.	do.[12]		555	520	[20] (24)	
96. Phenols, milligrams per liter.	Colorimetric, (4AAP)	241	582	545		
97. Phosphorus (elemental), milligrams per liter.	Gas chromatography [24]					
98. Phosphorus; total (as P), milligrams per liter.	Persulfate digestion followed by manual or automated ascorbic acid reduction.	249 256	476, 481 624	384	133	[3] (621)
RADIOLOGICAL						
99. Alpha—Total, pCi per liter.	Proportional or scintillation counter.		648	591	[11] [25](75+78)	
100. Alpha—Counting error, pCi per liter.	do		648	594	[25] (79)	
101. Beta—Total, pCi per liter.	Proportional counter		648	601	[11] [25](75+78)	
102. Beta—Counting error, pCi per liter.	do		648	603	[25] (79)	
103. (a) Radium—Total, pCi per liter.	do		661	661		
(b) ^{226}Ra, pCi per liter	Scintillation counter		667		[25] (81)	
RESIDUE						
104. Total, milligrams per liter	Gravimetric, 103 to 105° C	270	91			
105. Total dissolved (filterable), milligrams per liter.	Glass fiber filtration, 180° C.	266	92			
106. Total suspended (nonfilterable), milligrams per liter.	Glass fiber filtration, 103 to 105° C.	268	94			
107. Settleable, milliliters per liter or milligrams per liter.	Volumetric or gravimetric		95			
108. Total volatile, milligrams per liter.	Gravimetric 550° C	272	95			
109. Specific conductance, micromhos per centimeter at 25° C.	Wheatstone bridge conductimetry.	275	71	120	148	[3] (606)
110. Sulfate (as SO_4), milligrams per liter.	Gravimetric; turbidimetric; or automated colorimetric (barium chloranilate).	277 279	493 496	424 425		[3] (624) [3] (622)
111. Sulfide (as S), milligrams per liter.	Titrimetric—Iodine for levels greater than 1 mg per liter; Methylene blue photometric.	284	505 503		154	
112. Sulfite (as SO_3), milligrams per liter.	Titrimetric, iodine-iodate	285	508	435		
113. Surfactants, milligrams per liter.	Colorimetric (Methylene blue).	157	600	494	[20] (11)	
114. Temperature, degrees C	Calibrated glass or electrometric thermometer.	286	125		[20] (21)	
115. Turbidity, NTU	Nephelometric.	295	132	223	156	

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[1] Recommendations for sampling and preservation of samples according to parameter measured may be found in "Methods for Chemical Analysis of Water and Wastes, 1974" U.S. Environmental Protection Agency, table 2, pp. viii-xii.

[2] All page references for USGS methods, unless otherwise noted, are to Brown, E., Skougstad, M. W., and Fishman, M. J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A1, (1970).

[3] EPA comparable method may be found on indicated page of "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

[4] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[5] The method used must be specified.

[6] The 5 tube MPN is used.

[7] Slack, K. V. and others, "Methods for Collection and Analysis of Aquatic Biological and Mircobiological Samples: U.S. Geological Survey Techniques of Water-Resources Inv. book 5, ch. A4 (1973)."

[8] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

[9] Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[10] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, N.Y. 10018.

[11] Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters," (1976) open-file report 76-177.

[12] Procedures for pentachlorophenol, chlorinated organic compounds ,and pesticides can be obtained from the Environmental Monitoring and Support Lboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[13] Color method (ADMI procedure) available from Environmental Monitoring and Support Lbaoratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[14] For samples suspected of having thiocyanate interference, magnesium chloride is used as the digestion catalyst. In the approved test procedure for cyanides, the recommended catalysts are replaced with 20 ml of a solution of 510 g/l magnesium chloride ($MgCl_2 \cdot 6H_2O$). This substitution will eliminate thiocyanate interference for both total cyanide and cyanide amendable to chlorination measurements.

[15] For the determination of total metals the sample is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through preciptation, a less vigorous treatment is recommended as given on p. 83 (4.1.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired the procedure on p. 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, iridium, osmium, palladium, platinum, rhodium and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representitive aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO_3. Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3.) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

[16] As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

[17] Dissolved metals are defined as those constituents which will pass though a 0.45 μm membrane filter. A prefiltration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

[18] See "Atomic Absorption Newsletter," vol. 13, 75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

[19] Method available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[20] Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/l 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended method is satisfactory.

[21] An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[22] A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on combustion-methane detection is also acceptable.

[23] Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water": U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3 (1972).

[24] R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," "Journal of Chromatography," vol. 47, No. 3, pp. 421-426, 1970.

[25] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only suspended. Therefore, the 2 results must be added together to obtain "total."

[26] Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation: U.S. Geological Survey Techniques of Water Resources Inv., book 1 (1975)."

4. In § 136.4, the second sentence of paragraph (c) is amended by deleting the word "subchapter" immediately following the phrase "procedure under this" and immediately preceding the word "shall" and replaced with the phrase "paragraph c;" and § 136.4 is amended by adding a new paragraph (d) to read as follows:

§ 136.4 Application for alternate test procedures.

* * * * *

(c) * * * Any application for an alternate test procedure under this paragraph (c) shall: * * *

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

§ 136.5 [Amended]

5. In § 136.5, paragraph (a) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "test procedure" and before the period that ends the paragraph.

6. In § 136.5, paragraph (b) is amended by inserting in the first sentence the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "such application" and immediately before the comma. The second sentence of paragraph (b) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "State Permit Program and to the Director of the" at the end of the sentence, and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

7. In § 136.5, paragraph (c) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the phrase "application for an alternate test procedure" and immediately before the comma; and by deleting the phrase "Methods Development and Quality Assurance Laboratory" immediately after the phrase "application to the Director of the" and immediately before the phrase "for review and recommendation" and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

8. In § 136.5, the first sentence of paragraph (d) is amended by inserting the phrase, "proposed by the responsible person or firm making the discharge," immediately after the phrase, "application for an alternate test procedure," and immediately before the comma.

The second sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "to the Regional Administrator by the Director of the," and immediately preceding the period ending the sentence and inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

The third sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "forwarded to the Director," and immediately before the second comma and by inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

9. Section 136.5 is amended by the addition of a new paragraph (e) to read as follows:

§ 136.5 Approval of alternate test procedures.

* * * * *

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the FEDERAL REGISTER not later than 15 days after such notification and determination is made.

[FR Doc.76-35082 Filed 11-30-76;8:45 am]

FEDERAL REGISTER, VOL. 41, NO. 232--WEDNESDAY, DECEMBER 1, 1976, pp. 52780-52786

PART 136--GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Amendment of Regulations; Corrections*

Page	Parameter Number	Correction or Addition
52783	62, Nickel	14th ed. Standard Methods - add "232" to page reference opposite the colorimetric method designation.
52784	89	Parameter and units, change "Nitrate" to "Nitrite"
52784	96, Phenols	Delete the present method designation, "Colorimetric, (4 AAP)," and replace it with the method designation, "Distillation followed by colorimetric, (4 AAP)."
52784	96, Phenols	14th ed. Standard Methods, change "582" to "574"

Amendment; Correction**

52784	106, Total Suspended Residue	In "Method" column, after "103 to 105°C," add: "Glass fiber filtration, 103-105°C, post-washing of residue." Opposite this entry, in the "other Approved Methods" column, add (537)[27] Then, on page 52785, add footnote 27 to read: "27. Standard Methods for the Examination of Water and Wastewater, 13th Edition (1971)."

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*Federal Register, Volume 42, No. 12, Tuesday, January 18, 1977, pp. 3306-3307

**Federal Register, Volume 42, No. 139, Wednesday, July 20, 1977, p. 37205

NPDES/CERTIFICATIONS
ALTERNATIVE TEST PROCEDURES FOR LIMITED USE

TOPIC: Application and approval for individual permit-holders to use alternate test procedures (alternate to those published in the Federal Register for NPDES/certification purposes) for specific discharges.

SOURCE: Federal Register, Title 40, Chapter I, Subchapter D, Part 136:
- Vol 38, No. 199, October 16, 1973
- Vol 41, No. 232, December 1, 1976

A. Application for Alternate Test Procedures

The responsible person or firm making the discharge applies to the EPA Regional Administrator (RA) in the Region where the discharge occurs, through the Director of the State agency having permit-issuing authority. If the state does not issue permits, the application is sent directly to the EPA RA. One must:

1. Provide identifying information, i.e., name, address, permit number, etc.
2. Identify the pollutant or parameter involved.
3. Provide justification for using alternate procedure rather than stipulated test.
4. Provide a detailed description of the proposed procedure with references regarding the applicability to the effluents in question.

B. Approval of Alternate Test Procedures

An EPA Regional Administrator (RA) has the final responsibility for approval.

1. The State Director conducts a technical and administrative review and forwards the application and his recommendation to the RA.
2. The RA conducts a technical and administrative review.
    a. If the State Director recommended rejection for scientific and technical reasons, the RA denies the application and sends a copy of the rejected application and his decision to the applicant, the State Director and to the Director of the Environmental Monitoring and Support Laboratory (EMSL).
    b. Before approving any application, the RA sends a copy of the application to the Director of EMSL for review and recommendation.
3. Prior to 90 days of receipt of the application by the RA, the Director of EMSL forwards to the RA a recommendation providing the scientific and other technical basis for acceptance or rejection of the application.
4. Within 90 days of receipt, the RA notifies the applicant and the appropriate State agency of approval or rejection, or else specifies additional information required for the decision.
5. A copy of all approval and rejection notifications are sent to EMSL for purposes of national coordination.

NPDES/CERTIFICATIONS
ALTERNATIVE TEST PROCEDURES FOR NATIONWIDE USE

TOPIC: Application and approval by any person, laboratory, manufacturer, etc., for nationwide use of an alternate test procedure (alternate to those published in the Federal Register for NPDES/Certification purposes).

SOURCE: Federal Register, Title 40, Chapter I, Subchapter D, Part 136, Vol 41, No. 232, December 1, 1976

A. Application for Alternate Test Procedures

Any interested person, laboratory, manufacturer, etc., applies to the Director, Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio 45268. One must:

1. Provide identifying information, i.e., name and address of the responsible person or firm.
2. Identify the pollutant(s) or parameter(s) involved.
3. Provide a detailed description of the proposed procedure with references regarding its applicability.
4. Provide comparability data (proposed procedure compared to procedure published in the Federal Register).

B. Approval of Alternate Test Procedures

The Administrator of the U.S. Environmental Protection Agency has the final responsibility for approval.

1. The Director of EMSL conducts a technical review.
2. Within 90 days of receipt, the Director of EMSL notifies the Administrator of his recommendation to approve or reject the application, or else returns the application for additional information required for the decision.
3. After notification of the EMSL recommendations, the Administrator determines whether or not the alternate test procedures meet the requirements set forth in the Federal Register, i.e., whether the procedures are to be approved or rejected.
4. Within fifteen days of the notification and determination, notice of the final decision is submitted to the Federal Register for publication.

federal register

[6560-01]

ENVIRONMENTAL PROTECTION AGENCY

[FRL 863-7]

APPROVAL OF ALTERNATE WATER POLLUTANT TESTING PROCEDURE

232

Chemical Oxygen Demand (COD)

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (FEDERAL REGISTER; Vol. 41, No. 233, Wednesday, December 1, 1976, pp. 52780-52786), the Oceanography International Corp. applied for approval of a new test procedure for the measurement of COD. The new procedure incorporates a modification of the digestion technique of the approved procedure in that it uses a semimicro volume of sample and reagents, sealed in a glass ampule, with reflux digestion performed in an oven.

After a through review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study and other information submitted by the applicant in accordance with § 136.5, the EPA has designated the Oceanography International COD procedure as an approved alternate procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (the EPA's regulation implementing the Freedom of Information Act) at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Oceanography International COD procedure is acceptable for use by any person required to use approved procedures under § 304(g) of the Federal Water Pollution Control Act Amendments of 1972. For such use, the procedure must be used in strict accordance with the methods descriptions. The approved methods descriptions and prepackaged reagent ampules are available for the standard, low value, and high chloride ampule COD procedures from Oceanography International Corp., P.O. Box 2980, 512 West Loop, College Station, Tex. 77840.

Additional information concerning this action may be obtained by writing to the Director, Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

Dated: March 2, 1978.

STEPHEN J. GAGE,
Acting Assistant Administrator for Research and Development.

[FR Doc. 78-5965 Filed 3-6-78; 8:45 am]

VOL. 43, NO. 45—TUESDAY, MARCH 7, 1978

[6560-01]

ENVIRONMENTAL PROTECTION AGENCY

[FRL 956-2]

APPROVAL OF ALTERNATE WATER POLLUTANT TESTING PROCEDURE

Hydrogen Ion (pH) and Ammonia

In accordance with section 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (FEDERAL REGISTER, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780-52786), the Technicon Instrument Corp. applied for apaproval of new test procedures for the measurement of hydrogen ion (pH) and ammonia. The automated pH electrode procedure is industrial method No. 378-75W/A, October 1976. The automated ammonia electrode procedure is industrial method No. 379-74W/E, February 19, 1976. Manual distillation of samples prior to ammonia measurement may be required as stated in footnote 4, FR 41, No. 232, Wednesday, December 1, 1976, p. 52785.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of information submitted by the applicant in accordance with § 136.5, the EPA has designated the Technicon methods as approved alternate procedures for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act) at the Environmental Monitoring and Support Laboratory, Cincinnati, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Technicon Instrument Corp. methods are acceptable for use by any person required to use procedures under § 304(h) of the Clean Water Act of 1977. For such use, the procedure must be used in strict accordance with the methods descriptions. The approved methods descriptions are available from Technicon Industrial System, Technicon Instrument Corp., Tarrytown, N.Y. 10591.

Additional information concerning this action may be obtained by writing to the Director, Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

Dated: August 23, 1978.

STEPHAN J. GAGE,
Assistant Administrator for Research and Development.

[FR Doc. 78-24286 Filed 8-28-78; 8:45 am]

FEDERAL REGISTER, VOL. 43, NO. 168—TUESDAY, AUGUST 29, 1978

Notice Approval of Alternate Water Pollutant Testing Procedure; Nitrite/Nitrogen

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants"(Federal Register, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Hach Chemical Company applied for approval of a new test procedure for the measurement of nitrite/nitrogen. The new Hach procedure is a diazotization method using NitriVer III nitrite reagent.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study and other information submitted by the applicant, in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate procedure for nationwise use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method descriptions. The approved method descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: April 24, 1979.

Stephen J. Gage,

Assistant Administrator for Research and Development.

[FRL 1216-6]

[FR Doc. 79-13321 Filed 4-30-79; 8:45 am]

BILLING CODE [illegible]

Federal Register / Vol. 44, No. 85

Tuesday, May 1, 1979

Approval of Alternate Water Pollutant Testing Procedure; Total Manganese

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (Federal Register, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Hach Chemical Company applied for approval of a new test procedure for the measurement of manganese. The new Hach procedure is a colorimetric procedure using prepackaged reagents based upon the same chemical reactions involved in the approved colorimetric procedure referenced in 40 CFR Part 136.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study, and other information submitted by the applicant in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method descriptions for manganese, periodate oxidation method, "Wastewater Analysis Handbook," Hach Chemical Company, p. 275 and p. 281.

For the analysis of total manganese, addition of acid and digestion as indicated in Footnote 15 of Federal Register, Vol. 41, No. 232, Wednesday, December 1, 1976, p. 52785, and § 4.1.4, p. 83 of "Methods for Chemical Analysis of Water and Waste," USEPA, 1974, is the required sample pretreatment. This approved alternate test procedure for total manganese is approved also for the determination of dissolved manganese after sample filtration through a 0.45 micron filter.

The approved method description and prepackaged reagents are available from the Director of Technical Information, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

Dated: June 4, 1979.

Tom Murphy,

Acting Assistant Administrator for Research and Development.

[FR Doc. 79-18308 Filed 6-13-79; 8:45 am]

BILLING CODE 6560-01-M

Federal Register / Vol. 44, No. 116

Thursday, June 14, 1979

[FRL 1470-2]

Approval of Alternate Water Pollutant Testing Procedure; Chemical Oxygen Demand (COD)

In accordance with § 136.5, 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" (FR Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Hach Chemical Company applied for approval of an alternate test procedure for the measurement of COD. The new procedure incorporates a modification of the digestion technique of the approved procedure in that it uses a semi-micro volume of sample and reagents in a capped vial with reflux digestion performed in an oven.

After a thorough review and evaluation by the U.S. Environment Protection Agency (EPA) of the results of comparability testing studies and other information submitted by the applicant, in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate test procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method description, Oxygen Demand, Chemical; Reaction Digestion Method, Water Analysis Handbook, pp. 2-166–2-170, 1979 edition.

The approved method descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: April 14, 1980.

Stephen J. Gage,

Assistant Administrator for Research and Development.

[FR Doc. 80-12172 Filed 4-18-80; 8:45 am]

BILLING CODE 6560-01-M

Federal Register / Vol. 45, No. 78

Monday, April 21, 1980

ENVIRONMENTAL PROTECTION AGENCY

[FRL 1502-2]

Approval of Alternate Water Pollutant Testing Procedures; Total and Dissolved Zinc and Copper

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants," the Hach Chemical Company applied for approval of alternate test procedures for the measurement of total and dissolved zinc and copper.

After a thorough review and evaluation by the United States Environmental Protection Agency (EPA) of the results of comparability testing studies and other information submitted by the applicant in accordance with § 136.5, the EPA has designated the Hach procedures as approved alternate test procedures for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As approved alternate test procedures, the Hach procedures are acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedures must be used in strict accordance with the method descriptions for copper: Bicinchoninate Method, Method 8506; and for zinc: Zincon Method, Method 8009. In making this approval, the EPA disassociates itself from any statements in the approved Hach Chemical Company procedures which do not specifically pertain to the determination of analytical equivalency.

The approved methods descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: May 21, 1980.

Stephen J. Gage,

Assistant Administrator for Research and Development.

[FR Doc. 80-16224 Filed 5-28-80; 8:45 am]

BILLING CODE 6560-01-M

Federal Register / Vol. 45, No. 105

Thursday, May 29, 1980

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
CINCINNATI, OHIO 45268

DATE: September 20, 1978

SUBJECT: Use of Chemical Test Kits for Compliance Monitoring

FROM: Dwight G. Ballinger, Director
Environmental Monitoring and Support
Laboratory - Cincinnati

Dwight G Ballinger

TO: Regional Quality Assurance Coordinators

A number of Regional Coordinators and Permit staff have received inquiries concerning the use of test kits for the parameters required by the Effluent Guidelines and Compliance Monitoring Sections of PL 92-500. This memorandum defines the position of EMSL on these test methods and contains recommendations for regional and state response to inquiries concerning acceptance.

Our interpretation of Section 304(g), implemented as "Guidelines Establishing Test Procedures for Analysis of Pollutants," in Federal Register, Dec. 1, 1976, is that the test kits are not equivalent to the procedures promulgated in the Federal Register and therefore are subject to the requirements governing alternate procedures. Therefore, it is necessary for the applicant to request the use of these test kits from the appropriate Regional Administrator as presented in the regulations. This recommendation is based upon the following factors:

SAMPLE PREPARATION

Most of the procedures selected for implementation of Section 304(g) are designed to measure the total constituent or element present in the sample or are specifically modified to determine a precisely defined form of the substance. The test kits considered herein generally do not utilize the necessary digestion or pretreatment required to measure the total constituent. In many cases, such as preliminary digestion with acids or distillations, these pretreatments cannot be performed satisfactorily under field conditions. Thus, the final result reported, when a kit is used, is nearly always less than the true value and acceptance of such data will lead to wrong interpretation of effluent loadings.

INTERFERENCES

It should be recognized that a majority of these test kit measurement techniques were developed for field use on domestic water supplies or relatively clean ambient streams and are not designed to provide for removal of interferences often encountered in municipal and industrial waste effluents.

MEASUREMENT SYSTEMS

In some cases the physical and chemical measurement principles differ from those employed in the reference methods. In addition, proprietary reagents of unknown composition are often provided with little or no information available on the reactions involved. Even though some of the tests are "based on Standard Methods," a number of factors rule out the acceptance of the test procedures as equivalent.

REAGENTS

In most cases the reagent concentrations are not the same as those described in the reference method. Volumes are generally pre-measured under unknown conditions and addition to the sample is often by means of inaccurate droppers or pipets. In short, the quality and quantity of reagents is not under the direct control of the analyst, as required by good analytical technique.

CALIBRATION AND COLOR MEASUREMENT

In most test kits a comparatively simple photometer is provided or available light is used to measure color intensity. Calibration scales are supplied by the manufacturer based upon factors developed under ideal conditions in his laboratory, and recalibration is difficult or not recommended. No provision is made for changes in reagent composition due to inadequate quality control in manufacturing or adverse storage conditions. The photometers usually available will not accept cells of sufficient path length to achieve the required sensitivity, and sealed standards incorporated in the kits are subject to changes in color with time. Finally, the band pass of these photometers is generally too wide for accurate measurement of the appropriate wavelength.

DATA REQUIREMENTS FOR ALTERNATE TEST PROCEDURES

When consideration of the above factors are not sufficient to reject an application for use of test kits, a request should be made for comparative data upon which to judge the applicability of the alternative procedures. The attached protocol is recommended in developing the necessary information. The regulations require that the alternate procedures be used on the waste being monitored and that application be made to the Regional Administrator or State Director having jurisdiction over the permit issuance.

It is recognized that under unusual conditions the applicant may have difficulty obtaining comparative data because of lack of laboratory facilities at remote locations. Even in these cases, the applicant should be urged to obtain adequate data by having the necessary work done at a base laboratory or on contract. The acceptance of alternate procedures without supporting laboratory results may significantly weaken the pollution control efforts intended by PL 92-500.

The use of field test kits, as with other alternate procedures, should be considered on a case-by-case basis, with judgment based on all of the factors involved. No blanket acceptance of test methods will be recommended unless a large volume of data have been accumulated clearly showing that the use of the specific alternate procedure on a wide variety of sample types will provide test results equivalent in precision and accuracy to the reference methods. When such data are available, the method will probably be incorporated in amendments to the listing of Dec. 1, 1976, making further substantiation unnecessary.

Attachment

cc: Walter G. Gilbert, Director
National Training & Operational Technology Center

Attachment

DATA REQUIREMENTS
NATIONWIDE APPROVAL OF ALTERNATE TEST PROCEDURES

1) Five industrial (discharge) sources identified by Standard Industrial Classification (SIC) code or five drinking water sources,

2) Six samples from each source.

3) Four replicate analyses each by the proposed and approved method.

Sources		Samples		Replicates		Methods		Total
5	X	6	X	4	X	2	=	240

DATA REQUIREMENTS
LIMITED - USE APPROVAL OF ALTERNATE TEST PROCEDURE

STATE OR REGIONAL USE

1) Five sources.

2) Three samples from each source.

3) Four replicate analyses each by the proposed and approved method.

Sources		Samples		Replicates		Methods		Total
5	X	3	X	4	X	2	=	120

DATA REQUIREMENTS
LIMITED - USE APPROVAL OF ALTERNATE TEST PROCEDURE

PERMIT HOLDER OR DRINKING WATER SYSTEM

1) Three samples from each source.

2) Four replicate analyses each by the proposed and approved method.

Sources		Samples		Replicates		Methods		Total
1	X	3	X	4	X	2	=	24

STATISTICAL PROTOCOL
APPROVAL OF ALTERNATE TEST PROCEDURES

1) Calculate basic statistics of mean and standard deviation.

2) Test for outliers.

3) Frequency counts and histogram to check distribution.

4) Cochrans test for equality among within-sample standard deviation.

5) F-test for equality of pooled within-sample variances.

6) T-test for equality of method means.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
CINCINNATI, OHIO 45268

DATE: September 20, 1978

SUBJECT: Use of "Prepared" Reagents in NPDES Compliance

FROM: Dwight G. Ballinger, Director Dwight G. Ballinger
Environmental Monitoring and Support
Laboratory - Cincinnati

TO: Regional Quality Assurance Coordinators

We encourage the use of pre-prepared solutions and standards by sewage treatment plant operators and others for their required compliance monitoring activities, providing such solutions have been prepared according to the reagents section of the approved methods cited in the Federal Register of December 1, 1976. We would, however, be opposed to the use of commercial reagents that are of unknown composition.

In allowing the use of known pre-prepared solutions and standards, we strongly recommend that the following quality control checks be observed to insure their validity:

1. Date all solutions upon receipt of shipment, store in separate, appropriate area, and observe stated shelf life.

2. Verify that solutions and standards are valid by initially checking them against a quality control check sample available from EMSL through the appropriate Regional QA Coordinator. These check samples are available for most of the common measurements required in the NPDES. They are shipped in a sealed vial as a sample concentrate with the actual composition being provided in a separately sealed envelope.

3. Verify that these solutions are stable on a routine basis by periodically comparing them against a quality control check sample or a standard from another source.

cc: Walter G. Gilbert, Director
National Training & Operational Technology Center

federal register

WEDNESDAY, DECEMBER 24, 1975

THE NATIONAL ARCHIVES OF THE UNITED STATES • 1934 •

PART IV:

ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

National Interim Primary Drinking Water Regulations

10-27

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

[FRL 464-7]

PART 141—NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

On March 14, 1975, the Environmental Protection Agency (EPA) proposed National Interim Primary Drinking Water Regulations pursuant to sections 1412, 1414, 1415, and 1450 of the Public Health Service Act ("the Act"), as amended by the Safe Drinking Water Act ("SDWA," Pub. L. 93-523), 40 FR 11990. EPA held public hearings on the proposed regulations in Boston, Chicago, San Francisco, and Washington during the month of April. Several thousand pages of comments on the proposed regulations were received and evaluated. In addition, the Agency has received comments and information on the proposed regulations from the National Drinking Water Advisory Council, the Secretary of Health, Education, and Welfare, and from numerous others during meetings with representatives of State agencies, public interest groups and others.

The regulations deal only with the basic legal requirements. Descriptive material will be provided in a guidance manual for use by public water systems and the States.

The purpose of this preamble to the final regulations is to summarise the most significant changes made in the proposed regulations as a result of comments received and the further consideration of available information. A more detailed discussion of the comments and of changes in the proposed regulations is attached as Appendix A.

WATER SYSTEMS COVERED

The Safe Drinking Water Act applies to each "public water system," which is defined in Section 1401(4) of the Act as "a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves at least twenty-five individuals." Privately owned as well as publicly owned systems are covered. Service "to the public" is interpreted by EPA to include factories and private housing developments. (See generally, House Report, pp. 16-17.)

The definition of "public water system" proposed in the Interim Primary Drinking Water Regulations sought to explain the meaning of the statutory reference to "regular" service. It was proposed to interpret this term as including service for as much as three months during the year. Because the proposed definition would have excluded many large campgrounds, lodges, and other public accommodations which serve large numbers of tourists but which are open for slightly less than three months each year, the definition in the final version covers systems serving an average of at least twenty-five individuals at least 60 days out of the year. The use of a minimum number of days rather than months also makes clear that a system may qualify as a public water system even if it is not open every day during a given month.

Once "public water system" has been defined, it is necessary to define the two major types of public water systems—those serving residents and those serving transients or intermittent users. The possible health effects of a contaminant in drinking water in many cases are quite different for a person drinking the water for a long period of time than for a person drinking the water only briefly or intermittently. Different regulatory considerations may in some cases apply to systems which serve residents as opposed to systems which serve transients or intermittent users. Accordingly, § 141.2(e) makes clear that all "public water systems" fall within either the category of "community water systems" or the category of "non-community water systems." To make clear which regulatory requirements apply to which type of system, the category covered is specifically indicated throughout the regulations.

The proposed regulations defined a "community water system" as "a public water system which serves a population of which 70 percent or greater are residents." Reliance in the proposed definition on the percentage of water system users who are residents would result in treating some fairly large resort communities with many year-round residents as non-community systems. Therefore, the definition of "community water system" has been changed to cover any system which serves at least 15 service connections used by year-round residents or serves at least 25 year-round residents.

SMALL COMMUNITY WATER SYSTEMS

Many community water systems in the country are quite small. Since it is the intention of the Act to provide basically the same level of health protection to residents of small communities as to residents of large cities, and since a number of advanced water treatment techniques are made feasible only by economies of scale, the cost of compliance with the requirements of the Act may pose a serious problem for many small communities. The regulations seek to recognize the financial problems of small communities by requiring more realistic monitoring for systems serving fewer than 1,000 persons. Variances and exemptions authorized by the Act can also assist in dealing with economic problems of small community systems in appropriate cases, at least temporarily. EPA will provide technical assistance on effective treatment techniques which can be used by small systems.

These methods of dealing with the financial problems of some small community systems may not be sufficient in specific instances to make compliance with all applicable regulatory requirements feasible. EPA is commencing a study of potential problems faced by small community systems in meeting applicable requirements under the Act and these regulations, and, if necessary, will make additional adjustments in the Interim Primary Drinking Water Regulations prior to their effective date.

NON-COMMUNITY SYSTEMS

"Non-community systems" are basically those systems which serve transients. They include hotels, motels, restaurants, campgrounds, service stations, and other public accommodations which have their own water system and which have at least 15 service connections or serve water to a daily average of at least 25 persons. Some schools, factories and churches are also included in this category. It is conservatively estimated that there are over 200,000 non-community water systems in the country. However, it should be recognized that while their number is large, they normally are not the principal source of water for the people they serve.

The regulations as proposed would have applied all maximum contaminant levels to non-community systems as well as to community systems. This approach failed to take into account the fact that the proposed maximum contaminant levels for organic chemicals and most inorganic chemicals were based on the potential health effects of long-term exposure. Those levels are not necessary to protect transients or intermittent users. Therefore, the final regulations provide that maximum contaminant levels for organic chemicals, and for inorganic chemicals other than nitrates, are not applicable to non-community systems. An exception was made for nitrates because they can have an adverse health effect on susceptible infants in a short period of time.

Even without monitoring for organic chemicals or most inorganic chemicals, in the initial stages of implementation of the drinking water regulations, monitoring results from tens of thousands of non-community systems could overwhelm laboratory capabilities and other resources. This could delay effective implementation of the regulations with respect to the community systems which provide the water which Americans drink every day. To avoid this result, non-community systems will be given two years after the effective date of the regulations to commence monitoring. In the meantime, non-community systems which already monitor their water are encouraged to continue to do so, and the States are encouraged to take appropriate measures to test or require monitoring for non-community systems that serve large numbers of people.

Of course, non-community systems which pose a threat to health should be dealt with as quickly as possible. The maximum contaminant levels applicable to non-community water systems therefore will take effect 18 months after promulgation, at the same time as levels applicable to community systems. Inspection and enforcement authority will apply to non-community systems at the same time as to community systems.

SANITARY SURVEYS

EPA encourages the States to conduct sanitary surveys on a systematic basis.

These on-site inspections of water systems are more effective in assuring safe water to the public than individual tests taken in the absence of sanitary surveys. The regulations provide that monitoring frequencies for coliform bacteria can be changed by the entity with primary enforcement responsibility for an individual non-community system. and in certain circumstances for an individual community system. based on the results of a sanitary survey

Maximum Contaminant Levels

Numerous comments were received by EPA on the substances selected for the establishment of maximum contaminant levels and on the levels chosen Congress anticipated that the initial Interim Primary Drinking Water Regulations would be based on the Public Health Service Standards of 1962. and this Congressional intent has been followed. Comments received on the various levels did not contain new data sufficient to require the establishment of levels different from those contained in the Public Health Service Standards.

Water Consumption

The maximum contaminant levels are based, directly or indirectly. on an assumed consumption of two liters of water per day. The same assumption was used in the 1962 Standards. This assumption has been challenged because of instances where much higher water consumption rates occur. EPA's justification for using the two-liter figure is that it already represents an above average water or water-based fluid intake. Moreover, while the factor of safety may be somewhat reduced when greater quantities of water are ingested, the maximum contaminant levels based on the two-liter figure provide substantial protection to virtually all consumers. If, as has been suggested, a water consumption rate of eight liters per day is used as the basis for maximum contaminant level. all of the proposed MCL's would have to be divided by four, greatly increasing the monitoring difficulties, and in some cases challenging the sensitivity of accepted analytical procedures. It could be expected, in such a case, that the maximum contaminant levels would be exceeded to a significant degree, and that specialized treatment techniques would be required to order that the contaminant levels would be reduced. The economic impact of a move in this direction would be enormous. It is not technically or economically feasible to base maximum contaminant levels on unusually high consumption rates.

Safety Factors

A question was raised about the fact that different safety factors are contained in various maximum contaminant levels. The levels are not intended to have a uniform safety factor, at least partly because the knowledge of and the nature of the health risks of the various contaminants vary widely. The levels set are the result of experience, evaluation of the available data. and professional judgment They have withstood the test of time and of professional review. They are being subjected to further review by the National Academy of Sciences in connection with development of data for the Revised Primary Drinking Water Regulations

MCL's Based on Temperature

A question was also raised as to whether ranges of maximum contaminant levels should be established on the basis of the climate in the area served by the public water system. as was done with fluoride EPA believes that the use of a temperature scale for fluoride is more appropriate than for other chemicals because of the studies available on the fluoride-temperature relationship and because there is a small margin with fluoride between beneficial levels and levels that cause adverse health effects

MCL's Deleted

Three proposed maximum contaminant levels have been eliminated in the final regulations because they are not justified by the available data. One of these is carbon chloroform extract (CCE), which is discussed separately below. The others are the proposed levels for the standard bacterial plate count and cyanide. In the case of the plate count, it is believed that the coliform limits contained in the regulations, combined with the turbidity maximum contaminant level, adequately deal with bacterial contamination. However, EPA continues to believe that the standard plate count is a valid indicator of bacteriological quality of drinking water, and recommends that it be used in appropriate cases in conjunction with the coliform tests as an operational tool.

The proposed maximum contaminant level for cyanide was eliminated because the possibility of cyanide contamination can be effectively addressed only by the use of emergency action, such as under Section 1431 of the Act. EPA's 1969 Community Water Supply Study did not reveal a single instance in which cyanide was present in a water system at a level greater than one-thousandth of the level at which cyanide is toxic to humans.

Available data indicate that cyanide will be present in water systems at toxic levels only in the event of an accident, such as a spill from a barge collision. Maximum contaminant levels are not the appropriate vehicle for dealing with such rare, accidental contamination.

Heptachor, heptachlor epoxide and chlordane have also been removed from the list of maximum contaminant levels at least temporarily in view of the pending cancellation and suspension proceedings under the Federal Insecticide, Fungicide and Rodenticide Act involving those pesticides. When the results of these proceedings are available, EPA will again consider whether maximum contaminant levels should be established for those three pesticides

Sodium and Sulfates

A number of comments were received on the potential health effects of sodium and sulfates The National Drinking Water Advisory Council has recommended that consideration be given to the monitoring of these constituents but has not recommended the adoption of maximum contaminant levels because available data do not support the adoption of any specific levels EPA has requested the National Academy of Sciences to include sodium and sulfates among the contaminants to be studied by NAS. and to include information on the health effects of sodium and sulfates in the report to be made by NAS in December 1976

Since a number of persons suffer from diseases which are influenced by dietary sodium intake and since there are others who wish to restrict their sodium intake it is desirable that the sodium content of drinking water be known. Those affected can, by knowing the sodium concentration in their drinking water, make adjustments to their diets or in extreme cases, seek alternative sources of water to be used for drinking and food preparation It is recommended that the States institute programs for regular monitoring of the sodium content of drinking water served to the public, and for informing physicians and consumers of the sodium concentration in drinking water.

A relatively high concentration of sulfate in drinking water has little or no known laxative effect on regular users of the water, but transcients using such water sometimes experience a laxative effect. It is recommended that the States institute monitoring programs for sulfates, and that transients be notified if the sulfate content of the water is high. Such notification should include an assessment of the possible physiological effects of consumption of the water

PCB's and Asbestos

An interagency comment expressed concern for asbestos and PCB's in the environment and noted the need for at least a monitoring requirement, if not for MCL's, for these contaminants. EPA is also concerned. but for the moment lacks sufficient evidence regarding analytical methods, health effects, or occurrence in the environment to establish MCL's. The Agency is conducting research and cooperating in research projects to develop criteria for establishing needed limits as quickly as possible. A monitoring study on a number of organic chemical contaminants, including PCB's. for which MCL's are not being established at this time. will be contained in an organic chemical monitoring regulation that is being promulgated with these regulations. Regarding asbestos, HEW and EPA are sponsoring a number of studies this year at an approximate cost of $16 million to establish health effects anayltical methods and occurrence

Point of Measurement

Other comments on maximum contaminant levels focused on the proposed requirement that such levels be tested at the consumer's tap. Concern was expressed over the inability of the public water system to control potential sources of contaminants which are under the control of the consumer.

The promulgated definition of "maximum contaminant level," § 141.2(d), retains the requirement that the maximum contaminant level be measured at the tap except in the case of turbidity, which should be measured at the point of entry to the distribution system. However, the definition has been expanded to make clear that contaminants added to the water by circumstances under the control of the consumer are not the responsibility of the supplier of water, unless the contaminants result from corrosion of piping and plumbing resulting from the quality of the water supplied. It should be noted, however, that this requirement should not be interpreted as to discourage local, aggressive cross connection control measures.

COLIFORM BACTERIA MCL'S

The promulgated MCL's for coliform bacteria are basically the 1962 Public Health Service Standards, with minor refinements and clarifications. However, further changes may be desirable. For example, the MCL's for the membrane filter analytical method do not resolve the question of how many coliform bacteria are assumed to be present in a single highly contaminated sample. Some laboratories assume an upper limit of 50, while others seek to continue to count individual bacteria to a level of 100 or even higher in a single sample. The upper limit assumed will affect the monthly average which is calculated to determine compliance with the MCL's.

Another question relating to the coliform bacteria MCL's is the matter of possible spurious positive samples. As the regulations are written, all routine samples taken to determine compliance with the MCL's must be counted, regardless of the results of analysis of any check samples that may be taken. The reason for this is that bacterial contamination is often intermittent or transient, and as a result negative check samples taken a day or more after a positive sample cannot demonstrate that the positive result was in error. It may be possible, however, to prescribe a means of dealing with spurious positive results without compromising the integrity of the MCL's.

A third question concerning the MCL's for coliform bacteria is the relationship of monthly averages of coliform bacteria levels to monthly percentages of positive samples. For example, the monthly average MCL for the membrane filter method is violated if the monthly average exceeds one coliform bacterium per sample. However, for purposes of determining whether the monthly-percentage-of-positive-samples MCL is violated, a sample is counted as positive only if it contains more than four coliform bacteria. Thus, it is possible, particularly when a relatively small number of samples is taken, for a system to fail the monthly average MCL even when no single sample taken during the month is out of compliance with the limit.

These and other questions concerning the coliform bacteria MCL's will be reviewed further by EPA. If review indicates that changes in the MCL's are desirable, those changes will be made as soon as possible but within 6 months, in time to take effect at the same time as the initial Interim Primary Drinking Water Regulations.

ORGANIC CHEMICALS

The proposed maximum contaminant levels for organic pesticides, other than the three which are the subject of cancellation and suspension proceedings, have been retained. It is anticipated that additional organic pesticides will be added to the regulations if surveys of pesticides in drinking water being conducted by EPA indicate that this is needed.

The proposed regulations also contained a maximum contaminant level for organic chemicals obtained by the carbon chloroform extract (CCE) method. It was anticipated by Congress that organic chemicals would be dealt with primarily in the Revised Primary Drinking Water Regulations because of the paucity of accurate data on the health effects of various organic chemicals, the large number of such chemicals, uncertainities over appropriate treatment techniques, and the need for additional information on the incidence of specific organic chemicals in drinking water supplies. EPA thought that the CCE standard might provide an appropriate means of dealing with organic chemicals as a class pending action on the Revised Primary Regulations.

The CCE standard was originally developed as a test for undesirable tastes and odors in drinking water. As concern developed over the health effects of organic chemicals, the possibility of using CCE as a health standard rather than an esthetic standard was considered.

As pointed out by numerous comments, CCE has many failings as an indicator of health effects of organic chemicals. To begin with, the test obtains information on only a fraction of the total amount of organic chemicals in the water sampled. Furthermore, there is serious question as to the reliability of CCE in identifying those organic chemicals which are most suspected of adverse health effects. In addition, there are no existing data on which a specific level for CCE can be established on a rational basis. To establish a maximum contaminant level under these circumstances would almost certainly do more harm than good. It could give a false sense of security to persons served by systems which are within the established level and a false sense of alarm to persons served by systems which exceed the level. It also would divert resources from efforts to find more effective ways of dealing with the organic chemicals problem.

EPA believes that the intelligent approach to the organic chemicals question is to move ahead as rapidly as possible along two fronts. First, EPA is adopting simultaneously with these regulations a Subpart E of Part 141, containing requirements for organic chemical monitoring pursuant to Sections 1445 and 1450 of the Act.

The regulations require that designated public water systems collect samples of raw and treated water for submission to EPA for organics analysis. EPA will analyze the samples for a number of broad organic parameters, including carbon chloroform extract (CCE), volatile and non-volatile total organic carbon (VTOC and NVTOC), total organic chlorine (TOCl), ultraviolet absorbancy, and fluorescence. In addition, monitoring will be required for probably 21 specific organic compounds. Selection of the specific compounds has been based on the occurrence or likelihood of occurrence in treated water, toxicity data and availability of practical analytical methods. Laboratory analyses will be used to evaluate the extent and nature of organic chemical contamination of drinking water, to evaluate the validity of the general organic parameters as surrogates for measures of harmful organic chemicals, and to determine whether there is an adequate basis for establishing maximum contaminant levels for specific organics or groups of organics.

Second, EPA is embarking on an intensive research program to find answers to the following four questions:

1. What are the effects of commonly occurring organic compounds on human health?

2. What analytical procedures should be used to monitor finished drinking water to assure that any Primary Drinking Water Regulations dealing with organics are met?

3. Because some of these organic compounds are formed during water treatment, what changes in treatment practices are required to minimize the formation of these compounds in treated water?

4. What treatment technology must be applied to reduce contaminant levels to concentrations that may be specified in the Primary Drinking Water Regulations?

This research will involve health-effects and epidemiological studies, investigations of analytical methodology, and pilot plant and field studies of organic removal unit processes. Some phases of the research are to be completed by the end of this year, while much of the remainder are to be completed within the next calendar year.

As soon as sufficient information is derived from the monitoring program and related research, the Interim Primary Drinking Water Regulations will be amended so that the organic chemicals problem can be dealt with without delay. The monitoring process will be completed within 1 year.

During the interim period, while satisfactory MCL's for organic contamination in drinking water are being developed, EPA will act in specific cases where appropriate to deal with organic contamination. If the EPA monitoring program reveals serious specific cases of contamination, EPA will work with State and local authorities to identify the source and nature of the problem and to take remedial action. EPA will also aid the States in identifying additional community water supplies that require analysis.

PUBLIC NOTICE

The public notice requirements proposed in § 141.32 did not distinguish between community and non-community public water systems. They would have required that public notice of non-compliance with applicable regulations be made by newspaper, in water bills, and by other media for all public water systems. These requirements are inappropriate and ineffective in the case of most non-community water systems. Those systems principally serve transients who do not receive water bills from the system and who probably are not exposed significantly to the local media. A more effective approach would be to require notice that can inform the transient before he drinks the system's water, and thereby both warn the transient and provide an incentive to the supplier of water to remedy the violation. Accordingly, Section 141.32 as adopted provides that in the case of non-community systems, the entity with primary enforcement responsibility shall require that notice be given in a form and manner that will insure that the public using the public water system is adequately informed.

The proposed public notice requirements also failed to distinguish between different types of violations of the Interim Primary Drinking Water Regulations. Since the urgency and importance of a notice varies according to the nature of the violation involved, § 141.32 as promulgated seeks to match the type of notice required with the type of violation involved. Written notice accompanying a water bill or other direct notice by mail is required for all violations of the regulations, including violations of monitoring requirements, and for the grant of a variance or exemption. In addition, notice by newspaper and notification to radio and television stations is required whenever a maximum contaminant level is exceeded, or when the entity with primary enforcement responsibility requires such broader notice.

QUALITY CONTROL AND TESTING PROCEDURES

Section 1401(1) of the Act defines "primary drinking water regulation" to include "quality control and testing procedures." The promulgated regulations include testing requirements for each maximum contaminant level, including check samples and special samples in appropriate cases. The regulations also specify the procedures to be followed in analyzing samples for each of the maximum contaminant levels. These procedures will be updated from time to time as advances are made in analytical methods. For example, references to "Standard Methods for the Examination of Water and Wastewater" are to the current, 13th. edition, but these references will be changed to cite the 14th edition when it is available in the near future.

A key element of quality control for public water systems is accurate laboratory analysis. Section 141.28 of the regulations provides that analyses conducted for the purpose of determining compliance with maximum contaminant levels must be conducted by a laboratory approved by the entity with primary enforcement responsibility. EPA will develop as soon as possible, in cooperation with the States and other interested parties, criteria and procedures for laboratory certification. A State with primary enforcement responsibility will have a laboratory certified by EPA pursuant to the prescribed criteria and procedures, and in turn will certify laboratories within the State.

Record-keeping requirements and reports to the State also will assist in quality control efforts.

RECORD-KEEPING

Adequate record-keeping is necessary for the proper operation and administration of a public water system. It is also important for providing information to the public, providing appropriate data for inspection and enforcement activities and providing information on which future regulations can be based. Accordingly, a new § 141.33 has been added to the regulations to require that each public water system maintain records of sample analyses and of actions to correct violations of the Primary Drinking Water Regulations.

ECONOMIC AND COST ANALYSIS

A comprehensive economics study has been made of the Interim Primary Drinking Water Regulations. This study estimates the costs of the regulations, evaluates the potential economic impact, and considers possible material and labor shortages. The results of this analysis are summarized here.

Total investment costs to community water systems to achieve compliance with these regulations are estimated to be between $1,050 and $1,765 million. It is estimated that non-community systems will invest an additional $24 million. The range of the estimate is due to uncertainty as to the design flow that will be used in installing treatment facilities. Systems not in compliance will have to consider sizing their new components to reflect average daily flow conditions, or maximum daily flow conditions in cases where system storage is not adequate.

This investment will be spread over several years. Investor-owned systems will bear about one-fourth of these costs, and publicly-owned systems the remainder. It is not anticipated that systems will have difficulty financing these capital requirements.

In annual terms, national costs are expected to be within the following ranges:

	In millions
Capital costs	$146-247
Operations and maintenance	263-263
Monitoring (routine only)	17- 35
Total	$426-545

Although these aggregate figures are large, most water consumers will not be significantly affected. For those users in systems serving 10,000 persons or more, the average annual treatment cost per capita may increase from less than $1.00 for systems requiring disinfection and lead control, to between $15 to $35 for control of turbidity and heavy metal removal. For systems serving less than 100 persons, the average annual per capita costs of disinfection, lead control and fluoride/arsenic removal are estimated to be between $2.10 and $11.80. However, if turbidity control or heavy metal removal were required in a system of this size then costs are expected to range from $52 to $237 per year per capita. EPA is aware of the serious potential economic impact on users in these small systems. However, the legislative history specifies that the regulations should be based on costs that can be reasonably afforded by large metropolitan or regional systems. Further economic evaluation of these systems is being conducted, and realistic options for these small systems are being reviewed. Options that will be under consideration include less costly treatment technologies; formation of regional systems; and use of alternative water sources. Industrial and commercial users, whether providing their own water or using public systems, are not expected to be significantly affected by these regulations.

Possible constraints to the implementation of the interim primary regulations were examined. Although there will be an increase in demand for chemicals, manpower, laboratories, and construction of treatment facilities, it is not anticipated that any of these factors will be a serious obstacle to implementation of these regulations over a reasonable time frame.

For the reasons given above, Chapter 40 of the Code of Federal Regulations is hereby amended by the addition of the following new Part 141. These regulations will take effect 18 months after promulgation.

(It is hereby certified that the economic and inflationary impacts of these regulations have been carefully evaluated in accordance with Executive Order 11821)

Dated: December 10, 1975.

RUSSELL E. TRAIN,
Administrator

Subpart A—General

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141.31 Reporting requirements.
141.32 Public notification of variances, exemptions, and non-compliance with regulations.
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AUTHORITY: Secs. 1412, 1414, 1445, and 1450 of the Public Health Service Act, 88 Stat. 1660 (42 U.S.C. 300g 1, 300g-3, 300j-4, and 300j-9).

Subpart A—General

§ 141.1 Applicability.

This part establishes primary drinking water regulations pursuant to section 1412 of the Public Health Service Act, as amended by the Safe Drinking Water Act (Pub. L. 93-523); and related regulations applicable to public water systems.

§ 141.2 Definitions.

As used in this part, the term:

(a) "Act" means the Public Health Service Act, as amended by the Safe Drinking Water Act, Pub. L. 93-523.

(b) "Contaminant" means any physical, chemical, biological, or radiological substance or matter in water.

(c) "Maximum contaminant level" means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of a public water system, except in the case of turbidity where the maximum permissible level is measured at the point of entry to the distribution system. Contaminants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

(d) "Person" means an individual, corporation, company, association, partnership, State, municipality, or Federal agency.

(e) "Public water system" means a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a "community water system" or a "non-community water system."

(i) "Community water system" means a public water system which serves at least 15 service connections used by year-round residents or regularly serves at least 25 year-round residents.

(ii) "Non-community water system" means a public water system that is not a community water system.

(f) "Sanitary survey" means an onsite review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

(g) "Standard sample" means the aliquot of finished drinking water that is examined for the presence of coliform bacteria.

(h) "State" means the agency of the State government which has jurisdiction over public water systems. During any period when a State does not have primary enforcement responsibility pursuant to Section 1413 of the Act, the term "State" means the Regional Administrator, U.S. Environmental Protection Agency.

(i) "Supplier of water" means any person who owns or operates a public water system.

§ 141.3 Coverage.

This part shall apply to each public water system, unless the public water system meets all of the following conditions:

(a) Consists only of distribution and storage facilities (and does not have any collection and treatment facilities);

(b) Obtains all of its water from, but is not owned or operated by, a public water system to which such regulations apply;

(c) Does not sell water to any person; and

(d) Is not a carrier which conveys passengers in interstate commerce.

§ 141.4 Variances and exemptions.

Variances or exemptions from certain provisions of these regulations may be granted pursuant to Sections 1415 and 1416 of the Act by the entity with primary enforcement responsibility. Provisions under Part 142, *National Interim Primary Drinking Water Regulations Implementation*—subpart E (Variances) and subpart F (Exemptions)—apply where EPA has primary enforcement responsibility.

§ 141.5 Siting requirements.

Before a person may enter into a financial commitment for or initiate construction of a new public water system or increase the capacity of an existing public water system, he shall notify the State and, to the extent practicable, avoid locating part or all of the new or expanded facility at a site which:

(a) Is subject to a significant risk from earthquakes, floods, fires or other disasters which could cause a breakdown of the public water system or a portion thereof; or

(b) Except for intake structures, is within the floodplain of a 100-year flood or is lower than any recorded high tide where appropriate records exist.

The U.S. Environmental Protection Agency will not seek to override land use decisions affecting public water systems siting which are made at the State or local government levels.

§ 141.6 Effective date.

The regulations set forth in this part shall take effect 18 months after the date of promulgation.

Subpart B—Maximum Contaminant Levels

§ 141.11 Maximum contaminant levels for inorganic chemicals.

(a) The maximum contaminant level for nitrate is applicable to both community water systems and non-community water systems. The levels for the other inorganic chemicals apply only to community water systems. Compliance with maximum contaminant levels for inorganic chemicals is calculated pursuant to § 141.23.

(b) The following are the maximum contaminant levels for inorganic chemicals other than fluoride:

Contaminant	*Level, milligrams per liter*
Arsenic	0.05
Barium	1.
Cadmium	0.010
Chromium	0.05
Lead	0.05
Mercury	0.002
Nitrate (as N)	10.
Selenium	0.01
Silver	0.05

(c) When the annual average of the maximum daily air temperatures for the location in which the community water system is situated is the following, the maximum contaminant levels for fluoride are:

Temperature Degrees Fahrenheit	Degrees Celsius	Level, milligrams per liter
53.7 and below	12.0 and below	2.4
53.8 to 58.3	12.1 to 14.6	2.2
58.4 to 63.8	14.7 to 17.6	2.0
63.9 to 70.6	17.7 to 21.4	1.8
70.7 to 79.2	21.5 to 26.2	1.6
79.3 to 90.5	26.3 to 32.5	1.4

§ 141.12 Maximum contaminant levels for organic chemicals.

The following are the maximum contaminant levels for organic chemicals. They apply only to community water systems. Compliance with maximum contaminant levels for organic chemicals is calculated pursuant to § 141.24.

	Level, milligrams per liter
(a) Chlorinated hydrocarbons:	
Endrin (1,2,3,4,10, 10-hexachloro-6,7-epoxy-1,4, 4a,5,6,7,8,8a-octahydro-1,4-endo, endo-5,8 - dimethano naphthalene).	0.0002
Lindane (1,2,3,4,5,6-hexachlorocyclohexane, gamma isomer).	0.004
Methoxychlor (1,1,1-Trichloro-2, 2 - bis [p-methoxyphenyl] ethane).	0.1
Toxaphene ($C_{10}H_{10}Cl_8$-Technical chlorinated camphene, 67-69 percent chlorine).	0.005

(b) Chlorophenoxys	
2,4 - D, (2,4-Dichlorophenoxyacetic acid)	0. 1
2,4,5-TP Silvex (2,4,5-Trichlorophenoxypropionic acid).	0. 01

§ 141.13 Maximum contaminant levels for turbidity.

The maximum contaminant levels for turbidity are applicable to both community water systems and non-community water systems using surface water sources in whole or in part. The maximum contaminant levels for turbidity in drinking water, measured at a representative entry point(s) to the distribution system, are:

(a) One turbidity unit (TU), as determined by a monthly average pursuant to § 141.22, except that five or fewer turbidity units may be allowed if the supplier of water can demonstrate to the State that the higher turbidity does not do any of the following:

(1) Interfere with disinfection;

(2) Prevent maintenance of an effective disinfectant agent throughout the distribution system; or

(3) Interfere with microbiological determinations.

(b) Five turbidity units based on an average for two consecutive days pursuant to § 141.22.

§ 141.14 Maximum microbiological contaminant levels.

The maximum contaminant levels for coliform bacteria, applicable to community water systems and non-community water systems, are as follows:

(a) When the membrane filter technique pursuant to § 141.21(a) is used, the number of coliform bacteria shall not exceed any of the following:

(1) One per 100 milliliters as the arithmetic mean of all samples examined per month pursuant to § 141.21 (b) or (c);

(2) Four per 100 milliliters in more than one sample when less than 20 are examined per month; or

(3) Four per 100 milliliters in more than five percent of the samples when 20 or more are examined per month.

(b)(1) When the fermentation tube method and 10 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

(i) more than 10 percent of the portions in any month pursuant to § 141.21 (b) or (c);

(ii) three or more portions in more than one sample when less than 20 samples are examined per month; or

(iii) three or more portions in more than five percent of the samples when 20 or more samples are examined per month.

(2) When the fermentation tube method and 100 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

(i) more than 60 percent of the portions in any month pursuant to § 141.21 (b) or (c);

(ii) five portions in more than one sample when less than five samples are examined per month; or

(iii) five portions in more than 20 percent of the samples when five or more samples are examined per month.

(c) For community or non-community systems that are required to sample at a rate of less than 4 per month, compliance with paragraphs (a), (b)(1), or (b)(2) of this section shall be based upon sampling during a 3 month period, except that, at the discretion of the State, compliance may be based upon sampling during a one-month period.

Subpart C—Monitoring and Analytical Requirements

§ 141.21 Microbiological contaminant sampling and analytical requirements.

(a) Suppliers of water for community water systems and non-community water systems shall analyze for coliform bacteria for the purpose of determining compliance with § 141.14. Analyses shall be conducted in accordance with the analytical recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 662–688, except that a standard sample size shall be employed. The standard sample used in the membrane filter procedure shall be 100 milliliters. The standard sample used in the 5 tube most probable number (MPN) procedure (fermentation tube method) shall be 5 times the standard portion. The standard portion is either 10 milliliters or 100 milliliters as described in § 141.14 (b) and (c). The samples shall be taken at points which are representative of the conditions within the distribution system.

(b) The supplier of water for a community water system shall take coliform density samples at regular time intervals, and in number proportionate to the population served by the system. In no event shall the frequency be less than as set forth below:

Population served:	*Minimum number of samples per month*
25 to 1,000	1
1,001 to 2,500	2
2,501 to 3,300	3
3,301 to 4,100	4
4,101 to 4,900	5
4,901 to 5,800	6
5,801 to 6,700	7
6,701 to 7,600	8
7,601 to 8,500	9
8,501 to 9,400	10
9,401 to 10,300	11
10,301 to 11,100	12
11,101 to 12,000	13
12,001 to 12,900	14
12,901 to 13,700	15
13,701 to 14,600	16
14,601 to 15,500	17
15,501 to 16,300	18
16,301 to 17,200	19
17,201 to 18,100	20
18,101 to 18,900	21
18,901 to 19 800	22
19,801 to 20,700	23
20,701 to 21,500	24
21,501 to 22,300	25
22,301 to 23,200	26
23 201 to 24,000	27
24,001 to 24,900	28
24,901 to 25,000	29
25,001 to 28,000	30
28,001 to 33,000	35
33,001 to 37,000	40
37,001 to 41,000	45
41,001 to 46,000	50
46.001 to 50,000	55
50,001 to 54,000	60
54,001 to 59,000	65
59,001 to 64,000	70
64,001 to 70,000	75
70,001 to 76,000	80
76,001 to 83,000	85
83,001 to 90,000	90
90,001 to 96,000	95
96,001 to 111,000	100
111,001 to 130,000	110
130,001 to 160,000	120
160,001 to 190,000	130
190,001 to 220,000	140
220,001 to 250,000	150
250,001 to 290,000	160
290,001 to 320,000	170
320,001 to 360,000	180
360,001 to 410,000	190
410,001 to 450,000	200
450,001 to 500,000	210
500,001 to 550,000	220
550,001 to 600,000	230
600 001 to 660,000	240
660,001 to 720,000	250
720,001 to 780,000	260
780,001 to 840,000	270
840,001 to 910,000	280
910,001 to 970,000	290
970,001 to 1,050,000	300
1,050,001 to 1,140,000	310
1,140,001 to 1,230,000	320
1,230,001 to 1,320,000	330
1,320,001 to 1,420,000	340
1,420,001 to 1,520,000	350
1,520,001 to 1,630,000	360
1,630,001 to 1,730,000	370
1,730,001 to 1,850,000	380
1,850,001 to 1,970,000	390
1,970,001 to 2,060,000	400
2,060,001 to 2,270,000	410
2,270,001 to 2,510,000	420
2,510,001 to 2,750,000	430
2,750,001 to 3,020,000	440
3,020,001 to 3,320,000	450
3,320,001 to 3,620,000	460
3,620,001 to 3,960,000	470
3,960,001 to 4,310,000	480
4,310,001 to 4,690,000	490
4,690,001 or more	500

Based on a history of no coliform bacterial contamination and on a sanitary survey by the State showing the water system to be supplied solely by a protected ground water source and free of sanitary defects, a community water system serving 25 to 1,000 persons, with written permission from the State, may reduce this sampling frequency except that in no case shall it be reduced to less than one per quarter.

(c) The supplier of water for a non-community water system shall sample for coliform bacteria in each calendar quarter during which the system provides water to the public. Such sampling shall begin within two years after the effective date of this part. If the State, on the basis of a sanitary survey, determines that some other frequency is more appropriate, that frequency shall be the frequency required under these regulations. Such frequency shall be confirmed or changed on the basis of subsequent surveys.

(d)(1) When the coliform bacteria in a single sample exceed four per 100 milliliters (§ 141.14(a)), at least two consecutive daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show less than one coliform bacterium per 100 milliliters.

(2) When coliform bacteria occur in three or more 10 ml portions of a single sample (§ 141.14(b)(1)), at least two consecutive daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show no positive tubes.

(3) When coliform bacteria occur in all five of the 100 ml portions of a single sample (§ 141.14(b)(2)), at least two daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show no positive tubes.

(4) The location at which the check samples were taken pursuant to paragraphs (d) (1), (2), or (3) of this section shall not be eliminated from future sampling without approval of the State. The results from all coliform bacterial analyses performed pursuant to this subpart, except those obtained from check samples and special purpose samples, shall be used to determine compliance with the maximum contaminant level for coliform bacteria as established in § 141.14. Check samples shall not be included in calculating the total number of samples taken each month to determine compliance with § 141.21 (b) or (c).

(e) When the presence of coliform bacteria in water taken from a particular sampling point has been confirmed by any check samples examined as directed in paragraphs (d) (1), (2), or (3) of this section, the supplier of water shall report to the State within 48 hours.

(f) When a maximum contaminant level set forth in paragraphs (a), (b) or (c) of § 141.14 is exceeded, the supplier of water shall report to the State and notify the public as prescribed in § 141.31 and § 141.32.

(g) Special purpose samples, such as those taken to determine whether disinfection practices following pipe placement, replacement, or repair have been sufficient, shall not be used to determine compliance with § 141.14 or § 141.21 (b) or (c).

(h) A supplier of water of a community water system or a non-community water system may, with the approval of the State and based upon a sanitary survey, substitute the use of chlorine residual monitoring for not more than 75 percent of the samples required to be taken by paragraph (b) of this section, *Provided*, That the supplier of water takes chlorine residual samples at points which are representative of the conditions within the distribution system at the frequency of at least four for each substituted microbiological sample. There shall be at least daily determinations of chlorine residual. When the supplier of water exercises the option provided in this paragraph (h) of this section, he shall maintain no less than 0.2 mg/1 free chlorine throughout the public water distribution system. When a particular sampling point has been shown to have a free chlorine residual less than 0.2 mg/1, the water at that location shall be retested as soon as practicable and in any event within one hour. If the original analysis is confirmed, this fact shall be reported to the State within 48 hours. Also, if the analysis is confirmed, a sample for coliform bacterial analysis must be collected from that sampling point as soon as practicable and preferably within one hour, and the results of such analysis reported to the State within 48 hours after the results are known to the supplier of water. Analyses for residual chlorine shall be made in accordance with "Standard Methods for the Examination of Water and Wastewater," 13th Ed., pp. 129-132. Compliance with the maximum contaminant levels for coliform bacteria shall be determined on the monthly mean or quarterly mean basis specified in § 141.14, including those samples taken as a result of failure to maintain the required chlorine residual level. The State may withdraw its approval of the use of chlorine residual substitution at any time.

§ 141.22 Turbidity sampling and analytical requirements.

(a) Samples shall be taken by suppliers of water for both community water systems and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.13. The measurement shall be made by the Nephelometric Method in accordance with the recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 350-353, or "Methods for Chemical Analysis of Water and Wastes," pp. 295-298, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(b) If the result of a turbidity analysis indicates that the maximum allowable limit has been exceeded, the sampling and measurement shall be confirmed by resampling as soon as practicable and preferably within one hour. If the repeat sample confirms that the maximum allowable limit has been exceeded, the supplier of water shall report to the State within 48 hours. The repeat sample shall be the sample used for the purpose of calculating the monthly average. If the monthly average of the daily samples exceeds the maximum allowable limit, or if the average of two samples taken on consecutive days exceeds 5 TU, the supplier of water shall report to the State and notify the public as directed in § 141.31 and § 141.32.

(c) Sampling for non-community water systems shall begin within two years after the effective date of this part.

(d) The requirements of this § 141.22 shall apply only to public water systems which use water obtained in whole or in part from surface sources.

§ 141.23 Inorganic chemical sampling and analytical requirements.

(a) Analyses for the purpose of determining compliance with § 141.11 are required as follows.

(1) Analyses for all community water systems utilizing surface water sources shall be completed within one year following the effective date of this part. These analyses shall be repeated at yearly intervals.

(2) Analyses for all community water systems utilizing only ground water sources shall be completed within two years following the effective date of this part. These analyses shall be repeated at three-year intervals.

(3) For non-community water systems, whether supplied by surface or ground water sources, analyses for nitrate shall be completed within two years following the effective date of this part. These analyses shall be repeated at intervals determined by the State.

(b) If the result of an analysis made pursuant to paragraph (a) indicates that the level of any contaminant listed in § 141.11 exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses at the same sampling point within one month.

(c) When the average of four analyses made pursuant to paragraph (b) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall notify the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(d) The provisions of paragraphs (b) and (c) of this section notwithstanding, compliance with the maximum contaminant level for nitrate shall be determined on the basis of the mean of two analyses. When a level exceeding the maximum contaminant level for nitrate is found, a second analysis shall be initiated within 24 hours, and if the mean of the two analyses exceeds the maximum contaminant level, the supplier of water shall report his findings to the State pursuant to § 141.31 and shall notify the public pursuant to § 141.32.

(e) For the initial analyses required by paragraph (a)(1), (2) or (3) of this section, data for surface waters acquired within one year prior to the effective date and data for ground waters acquired within 3 years prior to the effective date of this part may be substituted at the discretion of the State.

(f) Analyses conducted to determine compliance with § 141.11 shall be made in accordance with the following methods:

(1) Arsenic--Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 95-96, Environ-

mental Protection Agency, Office of Technology Transfer, Washington, D.C 20460, 1974.

(2) Barium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, or "Methods for Chemical Analysis of Water and Wastes," pp. 97–98. Environmental Protection Agency, Office of Technology Transfer, Washington, D.C 20460, 1974

(3) Cadmium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, or "Methods for Chemical Analysis of Water and Wastes," pp. 101–103. Environmental Protection Agency, Office of Technology Transfer, Washington, D.C 20460, 1974.

(4) Chromium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, or "Methods for Chemical Analysis of Water and Wastes," pp. 105–106, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(5) Lead—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, or "Methods for Chemical Analysis of Water and Wastes," pp. 112–113, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(6) Mercury—Flameless Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 118–126, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(7) Nitrate—Brucine Colorimetric Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 461–464, or Cadmium Reduction Method, "Methods for Chemical Analysis of Water and Wastes," pp. 201–206, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(8) Selenium—Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," p. 145. Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(9) Silver—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater", 13th Edition, pp. 210–215, or "Methods for Chemical Analysis of Water and Wastes", p. 146, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(10) Fluoride—Electrode Method, "Standard Methods for the Examination of Water and Wastewater", 13th Edition, pp. 172–174, or "Methods for Chemical Analysis of Water and Wastes," pp. 65–67, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974, or Colorimetric Method with Preliminary Distillation, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 171–172 and 174–176, or "Methods for Chemical Analysis of Water and Wastes," pp. 59–60, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

§ 141.24 Organic chemical sampling and analytical requirements.

(a) An analysis of substances for the purpose of determining compliance with § 141.12 shall be made as follows.

(1) For all community water systems utilizing surface water sources, analyses shall be completed within one year following the effective date of this part. Samples analyzed shall be collected during the period of the year designated by the State as the period when contamination by pesticides is most likely to occur. These analyses shall be repeated at intervals specified by the State but in no event less frequently than at three year intervals.

(2) For community water systems utilizing only ground water sources, analyses shall be completed by those systems specified by the State.

(b) If the result of an analysis made pursuant to paragraph (a) of this section indicates that the level of any contaminant listed in § 141.12 exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses within one month.

(c) When the average of four analyses made pursuant to paragraph (b) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall report to the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(d) For the initial analysis required by paragraph (a) (1) and (2) of this section, data for surface water acquired within one year prior to the effective date of this part and data for ground water acquired within three years prior to the effective date of this part may be substituted at the discretion of the State.

(e) Analyses made to determine compliance with § 141.12(a) shall be made in accordance with "Method for Organochlorine Pesticides in Industrial Effluents," MDQARL, Environmental Protection Agency, Cincinnati, Ohio, November 28, 1973.

(f) Analyses made to determine compliance with § 141.12(b) shall be conducted in accordance with "Methods for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents," MDQARL, Environmental Protection Agency, Cincinnati, Ohio, November 28, 1973.

§ 141.27 Alternative analytical techniques.

With the written permission of the State, concurred in by the Administrator of the U.S. Environmental Protection Agency, an alternative analytical technique may be employed. An alternative technique shall be acceptable only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any maximum contaminant level. The use of the alternative analytical technique shall not decrease the frequency of monitoring required by this part.

§ 141.28 Approved laboratories.

For the purpose of determining compliance with § 141.21 through § 141.27, samples may be considered only if they have been analyzed by a laboratory approved by the State except that measurements for turbidity and free chlorine residual may be performed by any person acceptable to the State.

§ 141.29 Monitoring of consecutive public water systems.

When a public water system supplies water to one or more other public water systems, the State may modify the monitoring requirements imposed by this part to the extent that the interconnection of the sysems jusifies treating them as a single system for monitoring purposes. Any modified monitoring shall be conducted pursuant to a schedule specified by the State and concurred in by the Administrator of the U.S. Environmental Protection Agency.

Subpart D—Reporting, Public Notification and Record Keeping

§ 141.31 Reporting requirements.

(a) Except where a shorter reporting period is specified in this part, the supplier of water shall report to the State within 40 days following a test, measurement or analysis required to be made by this part, the results of that test, measurement or analysis.

(b) The supplier of water shall report to the State within 48 hours the failure to comply with any primary drinking water regulation (including failure to comply with monitoring requirements) set forth in this part.

(c) The supplier of water is not required to report analytical results to the State in cases where a State laboratory performs the analysis and reports the results to the State office which would normally receive such notification from the supplier.

§ 141.32 Public notification.

(a) If a community water system fails to comply with an applicable maximum contaminant level established in Subpart B, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, or fails to perform any monitoring required pursuant to Section 1445 (a) of the Act, the supplier of water shall notify persons served by the system of the failure or grant by inclusion of a notice in the first set of water bills of the system issued after the failure or grant and in any event by written notice within three months. Such notice shall be repeated at least once every three months so long as the system's failure continues or the variance or exemption remains in effect. If the system issues water bills less frequently than quarterly, or does not issue water bills, the notice shall be made by or supplemented by another form of direct mail.

(b) If a community water system has failed to comply with an applicable maximum contaminant level, the supplier of water shall notify the public of such failure, in addition to the notification required by paragraph (a) of this section, as follows:

(1) By publication on not less than three consecutive days in a newspaper or newspapers of general circulation in the area served by the system. Such notice shall be completed within fourteen days after the supplier of water learns of the failure.

(2) By furnishing a copy of the notice to the radio and television stations serving the area served by the system. Such notice shall be furnished within seven days after the supplier of water learns of the failure.

(c) If the area served by a community water system is not served by a daily newspaper of general circulation, notification by newspaper required by paragraph (b) of this section shall instead be given by publication on three consecutive weeks in a weekly newspaper of general circulation serving the area. If no weekly or daily newspaper of general circulation serves the area, notice shall be given by posting the notice in post offices within the area served by the system.

(d) If a non-community water system fails to comply with an applicable maximum contaminant level established in Subpart B of this part, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirement of any schedule prescribed pursuant to a variance or exemption or fails to perform any monitoring required pursuant to Section 1445(a) of the Act, the supplier of water shall given notice of such failure or grant to the persons served by the system. The form and manner of such notice shall be prescribed by the State, and shall insure that the public using the system is adequately informed of the failure or grant.

(e) Notices given pursuant to this section shall be written in a manner reasonably designed to inform fully the users of the system. The notice shall be conspicuous and shall not use unduly technical language, unduly small print or other methods which would frustrate the purpose of the notice. The notice shall disclose all material facts regarding the subject including the nature of the problem and, when appropriate, a clear statement that a primary drinking water regulation has been violated and any preventive measures that should be taken by the public. Where appropriate, or where designated by the State, bilingual notice shall be given. Notices may include a balanced explanation of the significance or seriousness to the public health of the subject of the notice, a fair explanation of steps taken by the system to correct any problem and the results of any additional sampling.

(f) Notice to the public required by this section may be given by the State on behalf of the supplier of water.

(g) In any instance in which notification by mail is required by paragraph (a) of this section but notification by newspaper or to radio or television stations is not required by paragraph (b) of this section, the State may order the supplier of water to provide notification by newspaper and to radio and television stations when circumstances make more immediate or broader notice appropriate to protect the public health.

§ 141.33 Record maintenance.

Any owner or operator of a public water system subject to the provisions of this part shall retain on its premises or at a convenient location near its premises the following records:

(a) Records of bacteriological analyses made pursuant to this part shall be kept for not less than 5 years. Records of chemical analyses made pursuant to this part shall be kept for not less than 10 years. Actual laboratory reports may be kept, or data may be transferred to tabular summaries, provided that the following information is included:

(1) The date, place, and time of sampling, and the name of the person who collected the sample;

(2) Identification of the sample as to whether it was a routine distribution system sample, check sample, raw or process water sample or other special purpose sample;

(3) Date of analysis;

(4) Laboratory and person responsible for performing analysis;

(5) The analytical technique/method used; and

(6) The results of the analysis.

(b) Records of action taken by the system to correct violations of primary drinking water regulations shall be kept for a period not less than 3 years after the last action taken with respect to the particular violation involved.

(c) Copies of any written reports, summaries or communications relating to sanitary surveys of the system conducted by the system itself, by a private consultant, or by any local, State or Federal agency, shall be kept for a period not less than 10 years after completion of the sanitary survey involved.

(d) Records concerning a variance or exemption granted to the system shall be kept for a period ending not less than 5 years following the expiration of such variance or exemption.

FROM p. 57332:

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 141

[FRL 1535-7]

Interim Primary Drinking Water Regulations; Amendments

AGENCY: Environmental Protection Agency (EPA).

→ **ACTION:** Final rule.

FROM p. 57332:

EFFECTIVE DATE: These amendments to the regulations will be effective August 27, 1980 except that sodium monitoring and reporting, determination of the types of materials in distribution systems, and monitoring and reporting corrosivity characteristics will be effective 18 months following the date of promulgation. The sodium and corrosion requirements must be completed within 12 months following the effective date.

FROM p. 57344:

9. Amending § 141.22(a) to read as follows and adding (e):

§ 141.22 Turbidity sampling and analytical requirements.

(a) Samples shall be taken by suppliers of water for both community and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.13. If the State determines that a reduced sampling frequency in a non-community system will not pose a risk to public health, it can reduce the required sampling frequency. The option of reducing the turbidity frequency shall be permitted only in those public water systems that practice disinfection and which maintain an active residual disinfectant in the distribution system, and in those cases where the State has indicated in writing that no unreasonable risk to health existed under the circumstances of this option. The turbidity measurements shall be made by the Nephelometric Method in accordance with the recommendations set forth in "Standard Methods for Examination of Water and Wastewater," American Public Health Association, 14th Edition, pp. 132-134; or Method 180.1.1-Nephrometric Method.

* * * * *

(e) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.

10. Amending § 141.23(a)(3), adding (a)(4) and amending (f) (1) through (10) to read as follows:

§ 141.23 Inorganic chemical sampling and analytical requirements.

(a) * * *

(3) For non-community water systems, whether supplied by surface or ground sources, analyses for nitrate shall be completed by December 24, 1980. These analyses shall be repeated at intervals determined by the State.

(4) The State has the authority to determine compliance or initiate enforcement action based upon analytical results and other information compiled by their sanctioned representatives and agencies.

* * * * *

(f) * * *

(1) Arsenic—Method [1] 206.2, Atomic Absorption Furnace Technique; or Method [1] 206.3, or Method [4] D2972-78A, or Method [2] 301.A VII, pp. 159-162, or Method [3] I-1062-78, pp. 61-63, Atomic Absorption—Gaseous Hydride; or Method [1] 206.4, or Method [4] D-2972-78A, or Method [2] 404-A and 404-B(4), Spectrophotometric, Silver Diethyldithiocarbamate.

(2) Barium—Method [1] 208.1, or Method [2] 301-A IV, pp. 152-155, Atomic Absorption—Direct Aspiration; or Method [1] 208.2, Atomic Absorption Furnace Technique.

(3) Cadmium—Method [1] 213.1, or Method [4] 3557-78A or B, or Method [2] 301-A II or III, pp. 148-152, Atomic Absorption—Direct Aspiration; or Method [1] 213.2, Atomic Absorption Furnace Technique.

(4) Chromium—Method [1] 218.1, or Method [4] D-1687-77D, or Method [2] 301-A II or III, pp. 148-152, Atomic Absorption—Direct Aspiration; or Chromium—Method [1] 218.2, Atomic Absorption Furnace Technique.

(5) Lead—Method [1] 239.1, or Method [4] D-3559-78A or B, or Method [2] 301-A II or III, pp. 148-152, Atomic Absorption—Direct Aspiration; or Method [1] 239.2, Atomic Absorption Furnace Technique.

(6) Mercury—Method [1] 245.1, or Method [4] D-3223-79, or Method [2] 301-A VI, pp. 156-159, Manual Cold Vapor Technique; or Method [1] 245.2, Automated Cold Vapor Technique.

(7) Nitrate—Method [1] 352.1, or Method [4] D-992-71, or Method [2] 419-D, pp. 427-429, Colorimetric Brucine; or Method [1] 353.3, or Method [4] D-3867-79B, or Method [2] 419-C, pp. 423-427, Spectrometric, Cadmium Reduction; Method [1] 353.1, Automated Hydrazine Reduction; or Method [1] 353.2, or Method [4] D-3867-79A, or Method [2] 605, pp. 620-624, Automated Cadmium Reduction.

(8) Selenium—Method [1] 270.2, Atomic Absorption Technique; or Method [1] 270.3; or Method [3] I-1667-78, pp. 237-239, or Method [4] D-3859-79, or Method [2] 301-A VII, pp. 159-162, Hydride Generation—Atomic Absorption Spectrophotometry.

(9) Silver—Method [1] 272.1, or Method [2] 301-A II, Atomic Absorption—Direct Aspiration; or Method [1] 272.2, Atomic Absorption Techniques Furnace Technique.

(10) Fluoride—Electrode Method, or SPADNS Method, Method [2] 414-B and C, pp. 391-394, or Method [1] 340.1, "Colorimetric SPADNS with Bellack Distillation," or Method [1] 340.2, "Potentimetric Ion Selective Electrode;" or ASTM Method [4] D1179-72; or Colorimetric Method with Preliminary Distillation, Method [2] 603, Automated Complexone Method (Alizarin Fluoride Blue) pp. 614-616; or Automated Electrode Method, "Fluoride in Water and Wastewater," Industrial Method #380-75WE, Technicon Industrial Systems, Tarrytown, New York 10591, February 1976, or "Fluoride in Water

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[1] "Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

[2] "Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.

[3] Techniques of Water—Resources Investigation of the United States Geological Survey, Chapter A-1, "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," Book 5, 1979, Stock #024-001-03177-9. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[4] Annual Book of ASTM Standards, part 31 Water, American Society for Testing and Materials, 1976, Race Street, Philadelphia, Pennsylvania 19103.

and Wastewater Industrial Method =129–71W," Technicon Industrial Systems, Tarrytown, New York 10591, December 1972; or Fluoride, Total, Colorimetic, Zirconium—Eriochrome Cyanine R Method [3]—I–3325–78, pp. 365–367.

11. Amending §141.24(a)(3), (e) and (f) to read as follows:

§ 141.24 Organic chemical sampling and analytical requirements.

(a) * * *

(3) The State has the authority to determine compliance or initiate enforcement action based upon analytical results and other information compiled by their sanctioned representatives and agencies.

* * * * *

(e) Analysis made to determine compliance with § 141.12(a) shall be made in accordance with "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268; or "Organochlorine Pesticides in Water," 1977 Annual Book of ASTM Standards, part 31, Water, Method D3088; or Method 509–A, pp. 555–565; [3] or Gas Chromatographic Methods for Analysis of Organic Substances in Water.[3] USGS, Book 5, Chapter A–5, pp. 24–39.

(f) Analysis made to determine compliance with § 141.12(b) shall be conducted in accordance with "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268; or "Chlorinated Phenoxy Acid Herbicides in Water," 1977 Annual Book of ASTM Standards, part 31, Method D3478; or Method 509–B, pp. 555–5692; [3] or Gas Chromatographic Methods for Analysis of Organic Substances in Water.[3] USGS, Book 5, Chapter A–3, pp. 24–39.

§ 141.25 [Amended]

12. Amending § 141.25 to add (e):

(e) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.

13. Amending § 141.27(a) to read as follows:

§ 141.27 Alternate analytical techniques.

(a) With the written permission of the State, concurred in by the Administrator of the U.S. EPA, an alternate analytical technique may be employed. An alternate technique shall be accepted only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any MCL. The use of the alternate analytical technique shall not decrease the frequency of monitoring required by this part.

14. Amending § 141.28 to read as follows:

§ 141.28 Approved laboratories.

(a) For the purpose of determining compliance with § 141.21 through § 141.27, samples may be considered only if they have been analyzed by a laboratory approved by the State except that measurements for turbidity, free chlorine residual, temperature and pH may be performed by any person acceptable to the State.

(b) Nothing in this Part shall be construed to preclude the State or any duly designated representative of the State from taking samples or from using the results from such samples to determine compliance by a supplier of water with the applicable requirements of this Part.

15. Amending § 141.31 (a) and (c) and adding paragraphs (d) and (e) to read as follows:

§ 141.31 Reporting requirements.

(a) Except where a shorter period is specified in this part, the supplier of water shall report to the State the results of any test measurement or analysis required by this part within (A) the first ten days following the month in which the result is received or (B) the first ten days following the end of the required monitoring period as stipulated by the State, whichever of these is shortest.

* * * * *

(d) The water supply system, within ten days of completion of each public notification required pursuant to § 141.32, shall submit to the State a representative copy of each type of notice distributed, published, posted, and/or made available to the persons served by the system and/or to the media.

(e) The water supply system shall submit to the State within the time stated in the request copies of any records required to be maintained under § 141.33 hereof or copies of any documents then in existence which the State or the Administrator is entitled to inspect pursuant to the authority of § 1445 of the Safe Drinking Water Act or the equivalent provisions of State law.

16. Amending § 141.32 (b)(3) and (d) to read as follows:

§ 141.32 Public notification.

* * * * *

(b) * * *

(3) Except that the requirements of this subsection (b) may be waived by the State if it determines that the violation has been corrected promptly after discovery, the cause of the violation has been eliminated, and there is no longer a risk to public health.

* * * * *

(d) If a non-community water system fails to comply with an applicable MCL established in Subpart B of this part, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable MCL, fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, or fails to perform any monitoring requirement pursuant to section 1445(a) of the Act, the supplier of water shall give notices by continuous posting of such failure or granting of a variance or exemption to the persons served by the system as long as the failure or granting of a variance or exemption continues. The form and manner for such notices shall be prescribed by the State and shall ensure that the public using the system is adequately informed of the failure or granting of the variance or exemption.

17. Amending Subpart E to read as follows:

Subpart E—Special Monitoring Regulations for Organic Chemicals and Otherwise Unregulated Contaminants

§ 141.41 Special monitoring for sodium.

(a) Suppliers of water for community public water systems shall collect and analyze one sample per plant at the entry point of the distribution system for the determination of sodium concentration levels; samples must be collected and analyzed annually for systems utilizing surface water sources in whole or in part, and at least every three years for systems utilizing solely ground water sources. The minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples. The supplier of water may be required by the State to collect and analyze water samples for sodium more frequently in locations where the sodium content is variable.

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[3] Techniques of Water—Resources Investigation of the United States Geological Survey, Chapter A–3, "Methods for Analysis of Organic Substances in Water," Book 5, 1972, Stock =2401–1227. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

(b) The supplier of water shall report to EPA and/or the State the results of the analyses for sodium within the first 10 days of the month following the month in which the sample results were received or within the first 10 days following the end of the required monitoring period as stipulated by the State, whichever of these is first. If more than annual sampling is required the supplier shall report the average sodium concentration within 10 days of the month following the month in which the analytical results of the last sample used for the annual average was received. The supplier of water shall not be required to report the results to EPA where the State has adopted this regulation and results are reported to the State. The supplier shall report the results to EPA where the State has not adopted this regulation.

(c) The supplier of water shall notify appropriate local and State public health officials of the sodium levels by written notice by direct mail within three months. A copy of each notice required to be provided by this paragraph shall be sent to EPA and/or the State within 10 days of its issuance. The supplier of water is not required to notify appropriate local and State public health officials of the sodium levels where the State provides such notices in lieu of the supplier.

(d) Analyses for sodium shall be performed by the flame photometric method in accordance with the procedures described in "Standard Methods for the Examination of Water and Wastewater," 14th Edition, pp. 250–253; or by Method 273.1, Atomic Absorption—Direct Aspiration or Method 273.2, Atomic Absorption—Graphite Furnace, in "Methods for Chemical Analysis of Water and Waste," EMSL, Cincinnati, EPA, 1979; or by Method D1428–64(a) in Annual Book of ASTM Standards, part 31, Water.

18. Adding a § 141.42 to read as follows:

§ 141.42 Special monitoring for corrosivity characteristics.

(a) Suppliers of water for community public water systems shall collect samples from a representative entry point to the water distribution system for the purpose of analysis to determine the corrosivity characteristics of the water.

(1) The supplier shall collect two samples per plant for analysis for each plant using surface water sources wholly or in part or more if required by the State; one during mid-winter and one during mid-summer. The supplier of the water shall collect one sample per plant for analysis for each plant using ground water sources or more if required by the State. The minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples.

(2) Determination of the corrosivity characteristics of the water shall include measurement of pH, calcium hardness, alkalinity, temperature, total dissolved solids (total filterable residue), and calculation of the Langelier Index in accordance with paragraph (c) below. The determination of corrosivity characteristics shall only include one round of sampling (two samples per plant for surface water and one sample per plant for ground water sources). However, States may require more frequent monitoring as appropriate. In addition, States have the discretion to require monitoring for additional parameters which may indicate corrosivity characteristics, such as sulfates and chlorides. In certain cases, the Aggressive Index, as described in paragraph (c), can be used instead of the Langelier Index; the supplier shall request in writing to the State and the State will make this determination.

(b) The supplier of water shall report to EPA and/or the State the results of the analyses for the corrosivity characteristics within the first 10 days of the month following the month in which the sample results were received. If more frequent sampling is required by the State, the supplier can accumulate the data and shall report each value within 10 days of the month following the month in which the analytical results of the last sample was received. The supplier of water shall not be required to report the results to EPA where the State has adopted this regulation and results are reported to the State.

(c) Analyses conducted to determine the corrosivity of the water shall be made in accordance to the following methods:

(1) Langelier Index—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 203, pp. 61–63.

(2) Aggressive Index—"AWWA Standard for Asbestos-Cement Pipe, 4 in. through 24 in. for Water and Other Liquids," AWWA C400–77, Revision of C400–75, AWWA, Denver, Colorado.

(3) Total Filtrable Residue—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 208B, pp. 92–93; or "Methods for Chemical Analysis of Water and Wastes," Method 160.1.

(4) Temperature—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 212, pp. 125–126.

(5) Calcium hardness—EDTA Titrimetric Method "Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 309B, pp. 202–206; or "Annual Book of ASTM Standards," Method D1126–67 (8).

(6) Alkalinity—Methyl Orange and paint pH 4.5. "Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 403, pp. 278–281; or "Annual Book of ASTM Standards," Method D1067–70B; or "Methods for Chemical Analysis of Water and Wastes," Method 310.1.

(7) pH—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 424, pp. 460–465; or "Methods for Chemical Analysis of Water and Wastes," Method 150.1; or "Annual Book of ASTM Standards," Method D129378 A or B.

(8) Chloride—Potentiometric Method. "Standard Methods for the Examination of Water and Wastewater," 14th Edition, p. 306.

(9) Sulfate—Turbidimetric Method. "Methods for Chemical Analysis of Water and Wastes," pp. 277–278, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 334–335, 14th Edition, pp. 496–498.

(d) Community water supply systems shall identify whether the following construction materials are present in their distribution system and report to the State:

- Lead from piping, solder, caulking, interior lining of distribution mains, alloys and home plumbing.
- Copper from piping and alloys, service lines, and home plumbing.
- Galvanized piping, service lines, and home plumbing.
- Ferrous piping materials such as cast iron and steel.
- Asbestos cement pipe.

In addition, States may require identification and reporting of other materials of construction present in distribution systems that may contribute contaminants to the drinking water, such as:

- Vinyl lined asbestos cement pipe.
- Coal tar lined pipes and tanks.

Appendix A—Response to Public Comments

ENC

Comments submitted to the Agency and statements presented at the public hearing in follows. These regulations will take effect January 19, 1981.

Dated: July 12, 1979.

Douglas M. Costle,
Administrator.

PART 143—NATIONAL SECONDARY DRINKING WATER REGULATIONS

Sec.
143.1 Purpose.
143.2 Definitions.
143.3 Secondary maximum contaminant levels.
143.4 Monitoring.

Authority: Section 1412(c) of the Safe Drinking Water Act, as amended (42 U.S.C. 300g–1(c))

§ 143.1 Purpose.

This part establishes National Secondary Drinking Water Regulations pursuant to Section 1412 of the Safe Drinking Water Act, as amended (42 U.S.C. 300g–1). These regulations control contaminants in drinking water that primarily affect the aesthetic qualities relating to the public acceptance of drinking water. At considerably higher concentrations of these contaminants, health implications may also exist as well as aesthetic degradation. The regulations are not Federally enforceable but are intended as guidelines for the States.

§ 143.2 Definitions.

(a) "Act" means the Safe Drinking Water Act as amended (42 U.S.C. 300f *et seq.*).

(b) "Contaminant" means any physical, chemical, biological, or radiological substance or matter in water.

(c) "Public water system" means a system for the provision to the public of piped water for human consumption, if such a system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a "community water system" or a "non-community water system."

(d) "State" means the agency of the State government which has jurisdiction over public water systems.

(e) "Supplier of water" means any person who owns or operates a public water system.

(f) "Secondary Maximum Contaminant Levels" means SMCLs which apply to public water systems and which, in the judgement of the Administrator, are requisite to protect the public welfare. The SMCL means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of public water system. Contamimants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

§ 143.3 Secondary Maximum Contaminant Levels.

The Secondary Maximum Contaminant Levels for public water systems are as follows:

Contaminant	Level
Chloride	250 mg/l.
Color	15 color units
Copper	1 mg/l.
Corrosivity	Noncorrosive
Foaming agents	0.5 mg/l.
Iron	0.3 mg/l.
Manganese	0.05 mg/l.
Odor	3 threshold odor number.
pH	6.5–8.5.
Sulfate	250 mg/l.
Total dissolved solids (TDS)	500 mg/l.
Zinc	5 mg/l.

These levels represent reasonable goals for drinking water quality. The States may establish higher or lower levels which may be appropriate dependent upon local conditions such as unavailability of alternate source waters or other compelling factors, provided that public health and welfare are not adversely affected.

§ 143.4 Monitoring.

(a) It is recommended that the parameters in these regulations should be monitored at intervals no less frequent than the monitoring performed for inorganic chemical contaminants listed in the National Interim Primary Drinking Water Regulations as applicable to community water systems. More frequent monitoring would be appropriate for specific parameters such as pH, color, odor or others under certain circumstances as directed by the State.

(b) Analyses conducted to determine compliance with § 143.3 should be made in accordance with the following methods:

(1) Chloride—Potentiometric Method, "Standard Methods for the Examination of Water and Wastewater," 14th Edition, p. 306.

(2) Color—Platinum-Cobalt Method, "Methods for Chemical Analysis of Water and Wastes," p. 36–38, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 160–162, 14th Edition, p. 64–66.

(3) Copper—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes", pp. 108–109, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

(4) Foaming Agents—Methylene Blue Method, "Methods for Chemical Analysis of Water and Wastes," pp. 157–158, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 339–342, 14th Edition, p. 600.

(5) Iron—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 110–111, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

(6) Manganese—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 116–117, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

(7) Odor—Consistent Series Method, "Methods for Chemical Analysis of Water and Wastes," pp. 287–294, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 248–254, 14th Edition, p. 75–82.

(8) pH—Glass Electrode Method, "Methods for Chemical Analysis of Water and Wastes," pp. 239–240, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 276–281, 14th Edition, pp. 460–465.

(9) Sulfate—Turbidimetric Method, "Methods for Chemical Analysis of Water and Wastes," pp. 277–278, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th

Edition, pp. 334–335, 14th Edition, p. 496–498.

(10) Total Dissolved Solids—Total Residue Methods. "Methods for Chemical Analysis of Water and Wastes," pp. 270–271, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 288–290, 14th Edition, p. 91–92.

(11) Zinc—Atomic Adsorption Method. "Methods for Chemical Analysis of Water and Wastes," pp. 155–156, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Laboratory Safety Practices

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 35 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participants must use correct laboratory safety practices during the laboratory sessions in this course and in their daily work situation.

V. ENTRY LEVEL BEHAVIOR:

A. At least two months experience performing any kind of chemical analyses in a laboratory

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participants will use proper safety procedures in carrying out all laboratory work.

B. Conditions: He/she will be given the training manual, a 30 minute discussion illustrating both equipment and practices required for safe conduct of laboratory procedures, and a 5 minute orientation to the location of safety devices in the student laboratory. Any safety equipment or facilities and any very specific, specialized safety precautions required for subsequent course laboratory assignments will be supplied.

C. Accepted Performance: Practice of the safety procedures during subsequent laboratory sessions.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in training manual, "Laboratory Safety Practices"

2. Forty slides, X-27: Laboratory Safety (See XI. Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the outline in the training manual and also the visual materials.

2. Prepare the lesson which includes an orientation to your laboratory facilities. Use the Sequencing below or your own organization of the material. Be prepared to give additional information such as examples, case histories, specific facts, etc.. Encourage the participants to share their experiences, too.

B. Sequencing:

Slides are Series X-27: Laboratory Safety.

1. Classroom

   a. Lab safety generally falls under the jurisdiction of one of these two; X-27, 1.

   b. At one time, USEPA considered withholding certification of drinking water analytical labs which had persistent, major safety problems; X-27, 2.

   c. A good lab safety program requires three elements; X-27, 3, 4, and 5.

   d. Example chain of command through which safety regulations are promulgated. Chance for confusion is great because of all the levels; X-27, 6.

   e. Introducing the next six slides which show possible lab accidents; X-27, 7.

   f. Possible lab accidents; X-27, 8 through 12, and 13 (the unforseen hazard).

   g. Introducing the next eleven slides which are photos of common lab safety errors; X-27, 14.

   h. Common lab safety errors; X-27, 15 through 22, 23 (possible back siphon, especially if there is a cross-connection with wastewater), 24 (can is overflowing and is not marked glass or paper), and 25 (no emergency phone numbers).

   i. Introducing the next thirteen slides which show common lab safety equipment; X-27, 26.

   j. Common lab safety equipment; X-27, 27 through 29 and 30 (other types of glasses/goggles are available), 31, 32 (first aid kits are not a substitute for professional help), 33 (Types A-Paper, B-Chemical, C-Electrical), 34 (siphon breaker) and 35 through 39.

   k. Purpose of a safety program is accident prevention, not just a program to assure prompt summons of an ambulance to carry someone out after an accident occurs; X-27, 40.

2. Laboratory - Point out the location of such safety devices as the fume hoods, eye-wash fountains, fire extinguishers, first aid kit, etc.

3. Laboratory - For each laboratory exercise, point out any special safety problems, e.g., use of toxic chemicals, handling of acid solutions, etc.

4. Subsequent laboratory sessions

   a. Point out any special safety precautions specific to the laboratory assignment.

   b. Observe individual performances regarding practice of the safety procedures.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visuals for classroom session

B. Safety equipment in laboratory for orientation

C. Specialized equipment for subsequent course laboratory sessions as required.

X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Forty slides, X-27: Laboratory Safety - See next pages.

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-27, LABORATORY SAFETY

PRINTING:

STATE/SAFETY HEALTH PROGRAM

OCCUPATIONAL SAFETY & HEALTH ACT

SLIDE 1

CARTOON OF SAFETY CERTIFICATE BEING DENIED BECAUSE OF HAZARDOUS LABORATORY PRACTICE

SLIDE 2

PRINTING:

SAFETY REQUIRES COMMON SENSE

SLIDE 3

PRINTING:

SAFETY COSTS MONEY

SLIDE 4

PRINTING:

SAFETY REQUIRES CHANGES IN ATTITUDE

SLIDE 5

PRINTING:

TOP MANAGEMENT → MIDDLE MANAGEMENT → PLANT SUPERVISOR → FOREMAN → PLANT WORKER

SLIDE 6

PRINTING:

LIFE-HEALTH ENDANGERING LABORATORY ACCIDENTS

SLIDE 7

CARTOON OF DEAD LAB WORKER WITH SANDWICH IN HAND AND A BOTTLE MARKED "TOXIC" NEARBY

SLIDE 8

CARTOON OF UNCONSCIOUS LAB WORKER WITH UNSTOPPERED BOTTLE MARKED "TOXIC-VOLATILE" NEARBY

SLIDE 9

SERIES X-27. LABORATORY SAFETY

CARTOON OF OPEN FLAMES ON A LAB TABLE WITH OTHER ITEMS (INCLUDING A WORKER'S LAB COAT) ON FIRE

SLIDE 10

DRAWING OF ACID DRIPPING FROM A TIPPED FLASK ONTO A LAB WORKER'S CLOTHING AND THE FLOOR

SLIDE 11

DRAWING OF A LAB WORKER WITH LIGHTED CIGARETTE WORKING NEXT TO A BOTTLE OF LIQUID MARKED "FLAMMABLE"

SLIDE 12

CARTOON DEPICTING HAZARDS OF PUBLIC RELATIONS - LAB WORKER BEING HIT WITH A BASEBALL BAT BY A WOMAN WHO IS SAYING, "MY WATER TASTES TERRIBLE."

SLIDE 13

PRINTING:
LABORATORY BOO-BOOS

SLIDE 14

PHOTO OF CLUTTERED WORK AREA

SLIDE 15

PHOTO OF LAB WORKER MOUTH-PIPETTING

SLIDE 16

PHOTO OF LAB WORKER POURING SULFURIC ACID WITH NONE OF THE USUAL PRECAUTIONS

SLIDE 17

PHOTO OF LAB WORKER CARRYING BOTTLE OF SULFURIC ACID WITHOUT A SAFETY CARRIER, ETC.

SLIDE 18

SERIES X-27, LABORATORY SAFETY

PHOTO OF LAB WORKER SMOKING A CIGARETTE WHILE TAKING A READING FROM A BURET

SLIDE 19

PHOTO OF LAB WORKER EATING AN APPLE WHILE WATCHING A BURET DURING A TITRATION

SLIDE 20

PHOTO OF LAB WORKER DRINKING FROM A BEAKER AT A LAB BENCH

SLIDE 21

PHOTO OF A BOTTLE OF "HEXANES" WITH CAP OFF, OUTSIDE A HOOD

SLIDE 22

PHOTO OF HOSE FROM A SINK FAUCET IMMERSED IN THE SINK

SLIDE 23

PHOTO OF A FILLED WASTE CAN WITH NO MARKING ABOUT GLASS OR PAPER, ETC.

SLIDE 24

PHOTO OF PHONE IN A LAB BUT NO EMERGENCY NUMBERS ARE POSTED

SLIDE 25

PRINTING:
COMMON SAFETY EQUIPMENT

SLIDE 26

PHOTO OF PIPET BULBS

SLIDE 27

SERIES X-27, LABORATORY SAFETY

PHOTO OF TWO PIPETORS

SLIDE 28

PHOTO OF LAB WORKER WEARING PLASTIC APRON

SLIDE 29

PHOTO OF PAIRS OF SAFETY GLASSES

SLIDE 30

PHOTO OF VOLATILE, FLAMMABLE CHEMICAL PROPERLY STORED IN A HOOD

SLIDE 31

PHOTO OF A FIRST AID KIT

SLIDE 32

PHOTO OF A FIRE EXTINGUISHER WITH AN INSPECTION TAG

SLIDE 33

PHOTO OF SINK AREA WITH NO GLASSWARE STANDING IN OR AROUND IT. THE HOSE FROM THE FAUCET COMES JUST TO THE TOP OF THE SINK.

SLIDE 34

PHOTO OF ACID BOTTLES IN TWO TYPES OF SAFETY CONTAINERS

SLIDE 35

PHOTO OF AN EXPLOSION-PROOF REFRIGERATOR

SLIDE 36

SERIES X-27, LABORATORY SAFETY

PHOTO TAKEN CLOSE-UP OF THE EXPLOSION-PROOF LABEL ON A REFRIGERATOR

SLIDE 37

PHOTO OF SIGN ON A REFRIGERATOR, "DO NOT STORE HAZARDOUS MATERIALS IN THIS UNIT".

SLIDE 38

PHOTO OF SIGNS ON A REFRIGERATOR, "DO NOT STORE FLAMMABLE MATERIALS IN THIS UNIT."

SLIDE 39

DRAWING OF AMBULANCE LEAVING TREATMENT PLANT

SLIDE 40 (LAST SLIDE)

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Analytical Techniques

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 20 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To carry out the volumetric analyses in the course, participants must correctly use volumetric glassware and titration assemblies.

V. ENTRY LEVEL BEHAVIOR:

A. Ability to use volumetric glassware

B. Ability to use titration assemblies

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will have reviewed correct techniques involved in basic laboratory operations and in using volumetric glassware and titration equipment.

B. Conditions: He/she will see silent films and be given numbers about correct techniques.

C. Accepted Performance: Subsequent satisfactory performance of the volumetric analyses included in the course.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Four segments of silent film showing an analyst demonstrating safety practices, general laboratory operations (like pouring chemicals from containers), techniques involved in cleaning and using pipets and a 3-valve bulb, and techniques involved in cleaning burets and carrying out a titration. These are available from Kalmia Co., Dept. C 1, Concord, Massachusetts 01742. Equivalent visual material is available from several other companies. Also, demonstration of the techniques is very effective.

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review films and/or equivalent visual materials and prepare commentary on techniques to review with participants. Personal experiences of Instructor and participants add interest and emphasis.

B. Sequencing:

1. Introduction - purpose of session

2. Show films and give commentary.

3. Encourage class sharing of pertinent experiences.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Films or equivalent visual materials.

X. IPW REAGENT REQUIREMENTS:

A. None

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Volumetric Analysis

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 55 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Participants should understand the principles of quantitation utilized to determine concentrations in water samples using volumetric analytical methodology as a prerequisite to the lessons on acidity, alkalinity, chlorine and hardness.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of quantitative analysis.

B. Ability to perform multiplication and division operations.

C. Laboratory experience in using volumetric glassware and titration assemblies.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will have reviewed the principles of quantitation involved in a volumetric analysis: analytical chemical reactions, indicators, volumes, standard solution concentrations, calculations.

B. Conditions: He/she will be given the training manual and a classroom discussion of the elements involved in this analytical method and in the calculation formulas utilized to obtain results.

C. Accepted Performance: Subsequent satisfactory performance of volumetric analyses of samples and ability to correctly use the appropriate calculation formula for acidity, alkalinity, hardness, and chlorine. He/she must also correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in training manual, "Volumetric Analysis of Water Quality"

2. Thirty slides, X-18: Volumetric Analysis (See XI. Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the topic outline in the training manual and the visual materials. See XI below.
2. Prepare the lesson using the sequencing below or your own organization of the material.

B. Sequencing:

Slides are series X-18: Volumetric Analysis
Participants can use the training outline as a reference for information on the slides.

1. Introduction - examples of water quality parameters that can be determined by volumetric analysis.
2. Titration Assembly, can use to give overview of topic - Slide 1
3. Titrations: definition, elements involved - Slides 2, 3, 4
4. Reactions involving solutions - Slides 5, 6, 7
   (Slide 5 - can define acid and base with this)
5. Completed reactions, items for discussion - Slide 8
6. Indicators - Slides 9, 10
7. Volume measurements for titrations - Slide 11
8. Standard Solutions: definition, terms - Slides 12, 13
9. Molar Solutions - Slides 14, 15, 16
10. Molecules react in definite ratio - Slide 17
11. Equivalent weights and normal solutions - Slides 18, 19, 20, 21
12. Volume relationship of normal solutions - Slide 22
13. Relating normality and grams - Slide 23
14. Finding normality - Slide 24
15. Finding grams - Slide 25
16. Application of formulas on slides 24 and 25 to acidity/alkalinity formula (Slide 26)
17. Primary standards - Slides 27, 28, 29
18. Preparation and storage of any standard solution - Slide 30

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual Materials

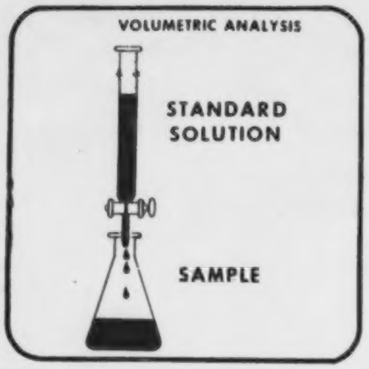
X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Thirty slides, X-18: Volumetric Analysis - See next pages.

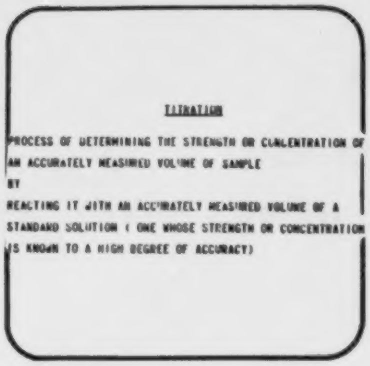
SERIES X-18: VOLUMETRIC ANALYSIS


VOLUMETRIC ANALYSIS

STANDARD SOLUTION

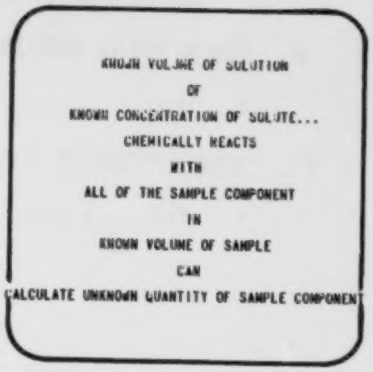
SAMPLE

SLIDE 1

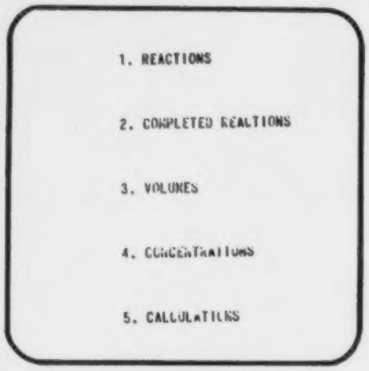

TITRATION

PROCESS OF DETERMINING THE STRENGTH OR CONCENTRATION OF AN ACCURATELY MEASURED VOLUME OF SAMPLE
BY
REACTING IT WITH AN ACCURATELY MEASURED VOLUME OF A STANDARD SOLUTION (ONE WHOSE STRENGTH OR CONCENTRATION IS KNOWN TO A HIGH DEGREE OF ACCURACY)

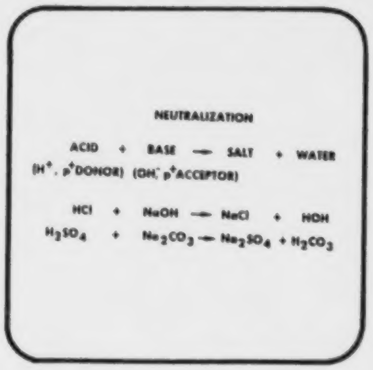
SLIDE 2


KNOWN VOLUME OF SOLUTION
OF
KNOWN CONCENTRATION OF SOLUTE...
CHEMICALLY REACTS
WITH
ALL OF THE SAMPLE COMPONENT
IN
KNOWN VOLUME OF SAMPLE
CAN
CALCULATE UNKNOWN QUANTITY OF SAMPLE COMPONENT

SLIDE 3


1. REACTIONS
2. COMPLETED REACTIONS
3. VOLUMES
4. CONCENTRATIONS
5. CALCULATIONS

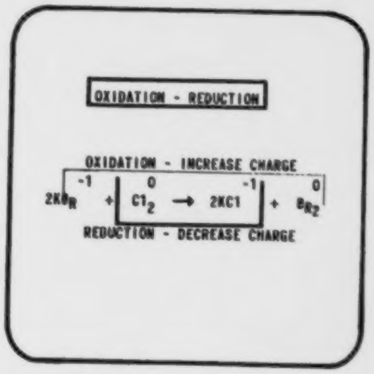
SLIDE 4


NEUTRALIZATION

ACID + BASE → SALT + WATER
(H^+, p^+DONOR) (OH^-, p^+ACCEPTOR)

$HCl + NaOH \rightarrow NaCl + HOH$
$H_2SO_4 + Na_2CO_3 \rightarrow Na_2SO_4 + H_2CO_3$

SLIDE 5

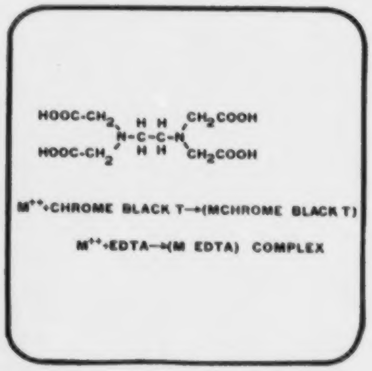

OXIDATION - REDUCTION

OXIDATION - INCREASE CHARGE

$$2K\overset{-1}{B_R} + \overset{0}{C1_2} \longrightarrow 2K\overset{-1}{C1} + \overset{0}{B_{R_2}}$$

REDUCTION - DECREASE CHARGE

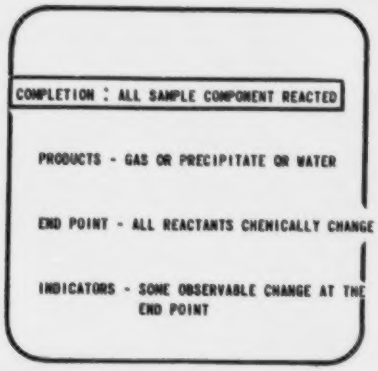
SLIDE 6


HOOC-CH2
HOOC-CH2
H H
N-C-C-N
H H
CH2COOH
CH2COOH

M^{++}+CHROME BLACK T→(MCHROME BLACK T)

M^{++}+EDTA→(M EDTA) COMPLEX

SLIDE 7

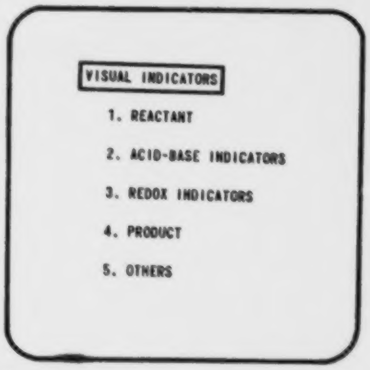

COMPLETION : ALL SAMPLE COMPONENT REACTED

PRODUCTS - GAS OR PRECIPITATE OR WATER

END POINT - ALL REACTANTS CHEMICALLY CHANGE

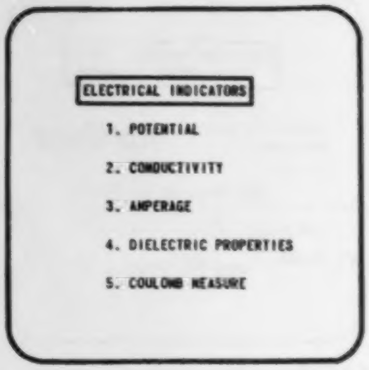
INDICATORS - SOME OBSERVABLE CHANGE AT THE END POINT

SLIDE 8


VISUAL INDICATORS
1. REACTANT
2. ACID-BASE INDICATORS
3. REDOX INDICATORS
4. PRODUCT
5. OTHERS

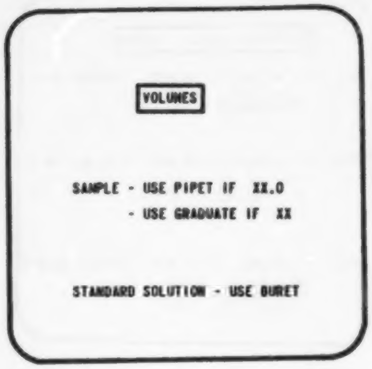
SLIDE 9

SERIES X-18: VOLUMETRIC ANALYSIS


ELECTRICAL INDICATORS

1. POTENTIAL
2. CONDUCTIVITY
3. AMPERAGE
4. DIELECTRIC PROPERTIES
5. COULOMB MEASURE

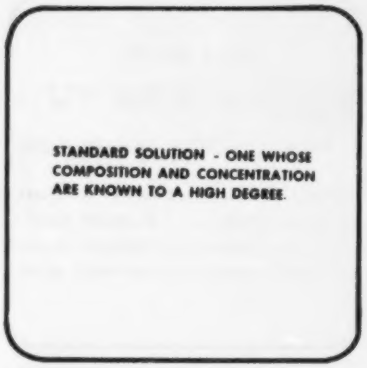
SLIDE 10


VOLUMES

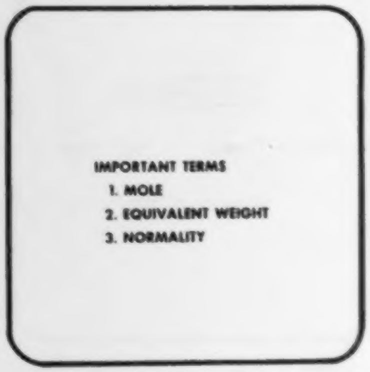
SAMPLE - USE PIPET IF XX.0
- USE GRADUATE IF XX

STANDARD SOLUTION - USE BURET

SLIDE 11

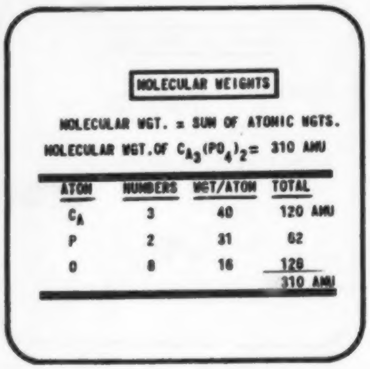

STANDARD SOLUTION - ONE WHOSE COMPOSITION AND CONCENTRATION ARE KNOWN TO A HIGH DEGREE.

SLIDE 12


IMPORTANT TERMS

1. MOLE
2. EQUIVALENT WEIGHT
3. NORMALITY

SLIDE 13

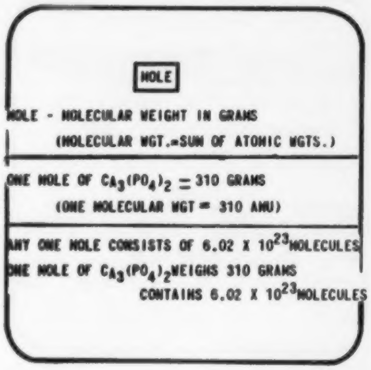

MOLECULAR WEIGHTS

MOLECULAR WGT. = SUM OF ATOMIC WGTS.

MOLECULAR WGT. OF $Ca_3(PO_4)_2$ = 310 AMU

ATOM	NUMBERS	WGT/ATOM	TOTAL
Ca	3	40	120 AMU
P	2	31	62
O	8	16	128
			310 AMU

SLIDE 14

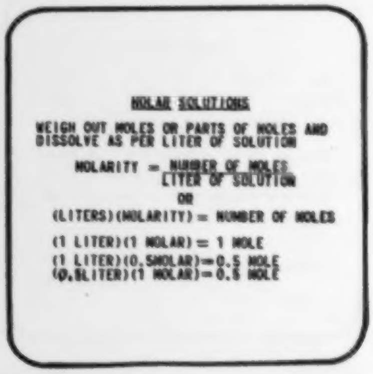

MOLE

MOLE - MOLECULAR WEIGHT IN GRAMS
(MOLECULAR WGT. = SUM OF ATOMIC WGTS.)

ONE MOLE OF $Ca_3(PO_4)_2$ = 310 GRAMS
(ONE MOLECULAR WGT = 310 AMU)

ANY ONE MOLE CONSISTS OF 6.02×10^{23} MOLECULES
ONE MOLE OF $Ca_3(PO_4)_2$ WEIGHS 310 GRAMS
CONTAINS 6.02×10^{23} MOLECULES

SLIDE 15


MOLAR SOLUTIONS

WEIGH OUT MOLES OR PARTS OF MOLES AND DISSOLVE AS PER LITER OF SOLUTION

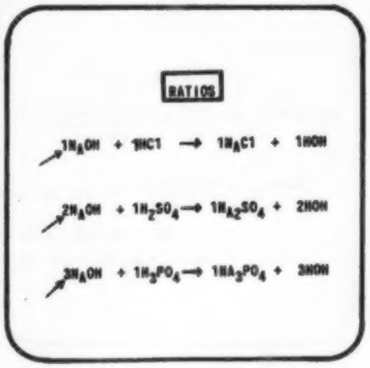
$$\text{MOLARITY} = \frac{\text{NUMBER OF MOLES}}{\text{LITER OF SOLUTION}}$$

OR

(LITERS)(MOLARITY) = NUMBER OF MOLES

(1 LITER)(1 MOLAR) = 1 MOLE
(1 LITER)(0.5 MOLAR) = 0.5 MOLE
(0.5 LITER)(1 MOLAR) = 0.5 MOLE

SLIDE 16

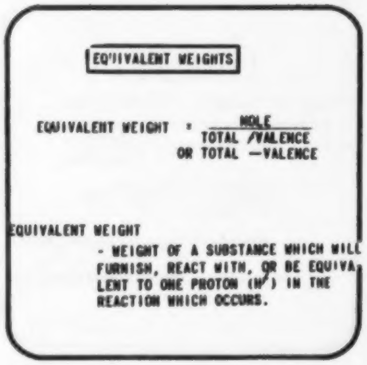

RATIOS

$1NaOH + 1HCl \rightarrow 1NaCl + 1HOH$

$2NaOH + 1H_2SO_4 \rightarrow 1Na_2SO_4 + 2HOH$

$3NaOH + 1H_3PO_4 \rightarrow 1Na_3PO_4 + 3HOH$

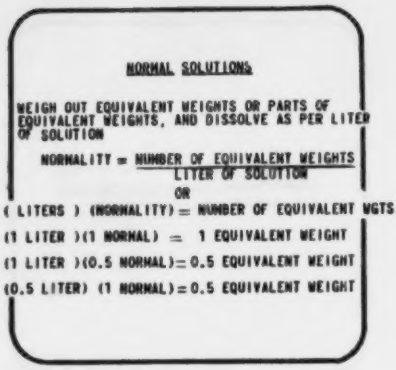
SLIDE 17


EQUIVALENT WEIGHTS

$$\text{EQUIVALENT WEIGHT} = \frac{\text{MOLE}}{\text{TOTAL +VALENCE}}$$
OR TOTAL —VALENCE

EQUIVALENT WEIGHT
- WEIGHT OF A SUBSTANCE WHICH WILL FURNISH, REACT WITH, OR BE EQUIVALENT TO ONE PROTON (H^+) IN THE REACTION WHICH OCCURS.

SLIDE 18



NORMAL SOLUTIONS

WEIGH OUT EQUIVALENT WEIGHTS OR PARTS OF EQUIVALENT WEIGHTS, AND DISSOLVE AS PER LITER OF SOLUTION

$$\text{NORMALITY} = \frac{\text{NUMBER OF EQUIVALENT WEIGHTS}}{\text{LITER OF SOLUTION}}$$

OR

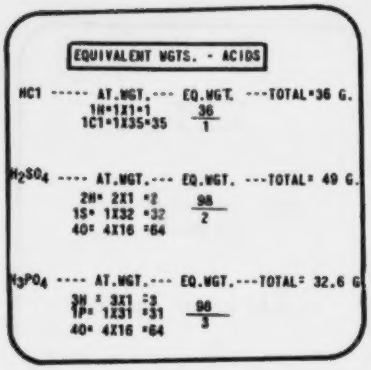
(LITERS) (NORMALITY) = NUMBER OF EQUIVALENT WGTS.

(1 LITER)(1 NORMAL) = 1 EQUIVALENT WEIGHT

(1 LITER)(0.5 NORMAL) = 0.5 EQUIVALENT WEIGHT

(0.5 LITER) (1 NORMAL) = 0.5 EQUIVALENT WEIGHT

SLIDE 19



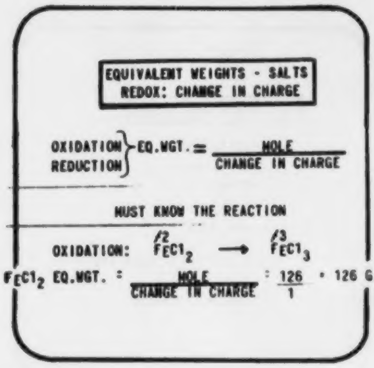
EQUIVALENT WGTS. - ACIDS

HC1 ----- AT.WGT.--- EQ.WGT. ---TOTAL=36 G.
1H=1X1=1
1C1=1X35=35
$\frac{36}{1}$

H_2SO_4 ---- AT.WGT.--- EQ.WGT. ---TOTAL= 49 G.
2H= 2X1 =2
1S= 1X32 =32
4O= 4X16 =64
$\frac{98}{2}$

H_3PO_4 ---- AT.WGT.--- EQ.WGT.---TOTAL= 32.6 G
3H = 3X1 =3
1P= 1X31 =31
4O= 4X16 =64
$\frac{98}{3}$

SLIDE 20



EQUIVALENT WEIGHTS - SALTS
REDOX: CHANGE IN CHARGE

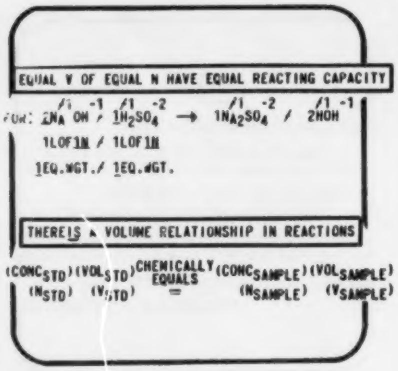
$$\left.\begin{matrix}\text{OXIDATION}\\\text{REDUCTION}\end{matrix}\right\}\text{EQ.WGT.} = \frac{\text{MOLE}}{\text{CHANGE IN CHARGE}}$$

MUST KNOW THE REACTION

OXIDATION: $\overset{+2}{\text{FeCl}_2} \longrightarrow \overset{+3}{\text{FeCl}_3}$

$$\text{FeCl}_2\ \text{EQ.WGT.} = \frac{\text{MOLE}}{\text{CHANGE IN CHARGE}} = \frac{126}{1} = 126\ \text{G.}$$

SLIDE 21



EQUAL V OF EQUAL N HAVE EQUAL REACTING CAPACITY

FOR: $\underline{2}\overset{+1}{\text{Na}}\overset{-1}{\text{OH}} + \underline{1}\overset{+1}{\text{H}_2}\overset{-2}{\text{SO}_4} \longrightarrow 1\overset{+1}{\text{Na}_2}\overset{-2}{\text{SO}_4} + 2\overset{+1}{\text{H}}\overset{-1}{\text{OH}}$

1LOF$\underline{1N}$ + 1LOF$\underline{1N}$

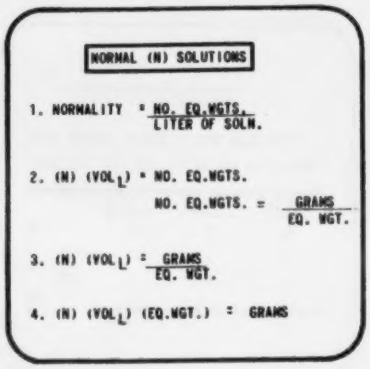
$\underline{1}$EQ.WGT.+ $\underline{1}$EQ.WGT.

THERE IS A VOLUME RELATIONSHIP IN REACTIONS

$$(\text{CONC}_{STD})(\text{VOL}_{STD}) \overset{\text{CHEMICALLY EQUALS}}{} (\text{CONC}_{SAMPLE})(\text{VOL}_{SAMPLE})$$

$$(N_{STD})\ (V_{STD}) = (N_{SAMPLE})\ (V_{SAMPLE})$$

SLIDE 22



NORMAL (N) SOLUTIONS

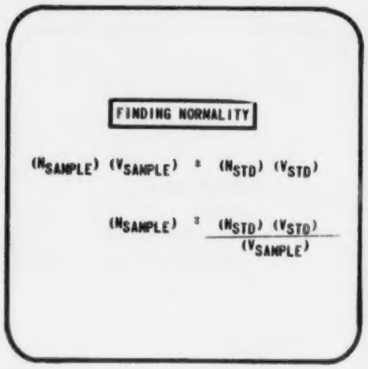
1. $\text{NORMALITY} = \frac{\text{NO. EQ.WGTS.}}{\text{LITER OF SOLN.}}$

2. $(N)\ (\text{VOL}_L) = \text{NO. EQ.WGTS.}$
$\text{NO. EQ.WGTS.} = \frac{\text{GRAMS}}{\text{EQ. WGT.}}$

3. $(N)\ (\text{VOL}_L) = \frac{\text{GRAMS}}{\text{EQ. WGT.}}$

4. $(N)\ (\text{VOL}_L)\ (\text{EQ.WGT.}) = \text{GRAMS}$

SLIDE 23

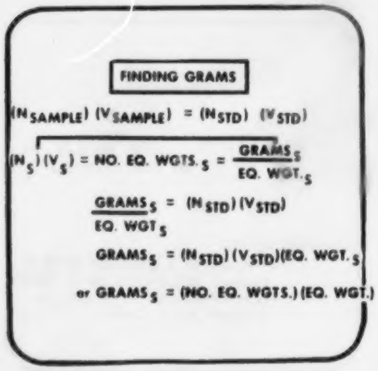


FINDING NORMALITY

$$(N_{SAMPLE})\ (V_{SAMPLE}) = (N_{STD})\ (V_{STD})$$

$$(N_{SAMPLE}) = \frac{(N_{STD})\ (V_{STD})}{(V_{SAMPLE})}$$

SLIDE 24



FINDING GRAMS

$$(N_{SAMPLE})\ (V_{SAMPLE}) = (N_{STD})\ (V_{STD})$$

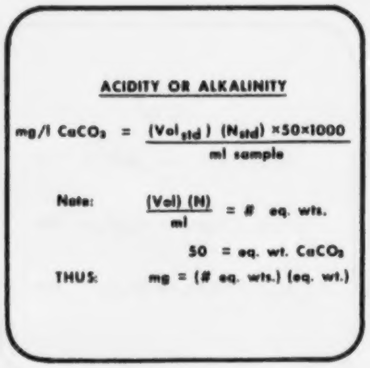
$$(N_S)(V_S) = \text{NO. EQ. WGTS.}_S = \frac{\text{GRAMS}_S}{\text{EQ. WGT.}_S}$$

$$\frac{\text{GRAMS}_S}{\text{EQ. WGT}_S} = (N_{STD})(V_{STD})$$

$$\text{GRAMS}_S = (N_{STD})(V_{STD})(\text{EQ. WGT.}_S)$$

$$\text{or GRAMS}_S = (\text{NO. EQ. WGTS.})(\text{EQ. WGT.})$$

SLIDE 25



ACIDITY OR ALKALINITY

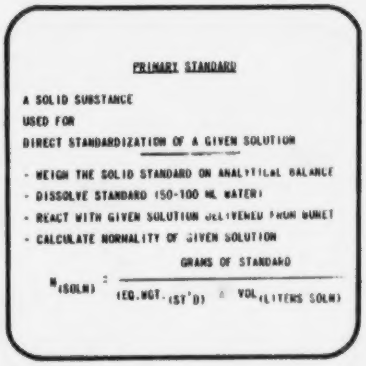
$$\text{mg/l CaCO}_3 = \frac{(\text{Vol}_{std})\ (N_{std}) \times 50 \times 1000}{\text{ml sample}}$$

Note: $\frac{(\text{Vol})\ (N)}{\text{ml}} = \#\ \text{eq. wts.}$

$50 = \text{eq. wt. CaCO}_3$

THUS: mg = (# eq. wts.) (eq. wt.)

SLIDE 26



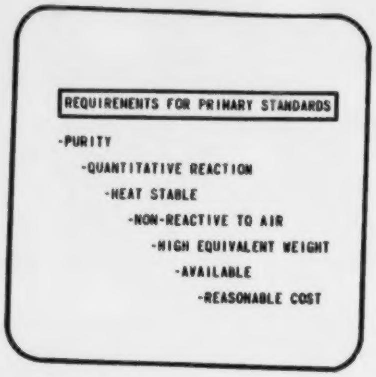
PRIMARY STANDARD

A SOLID SUBSTANCE
USED FOR
DIRECT STANDARDIZATION OF A GIVEN SOLUTION

- WEIGH THE SOLID STANDARD ON ANALYTICAL BALANCE
- DISSOLVE STANDARD (50-100 ML WATER)
- REACT WITH GIVEN SOLUTION DELIVERED FROM BURET
- CALCULATE NORMALITY OF GIVEN SOLUTION

$$N_{(SOLN)} = \frac{\text{GRAMS OF STANDARD}}{(\text{EQ.WGT.}_{(ST'D)} \times \text{VOL}_{(LITERS\ SOLN)})}$$

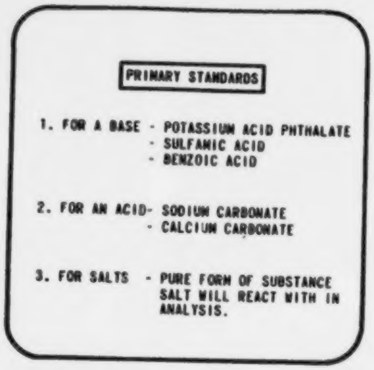
SLIDE 27



REQUIREMENTS FOR PRIMARY STANDARDS

-PURITY
-QUANTITATIVE REACTION
-HEAT STABLE
-NON-REACTIVE TO AIR
-HIGH EQUIVALENT WEIGHT
-AVAILABLE
-REASONABLE COST

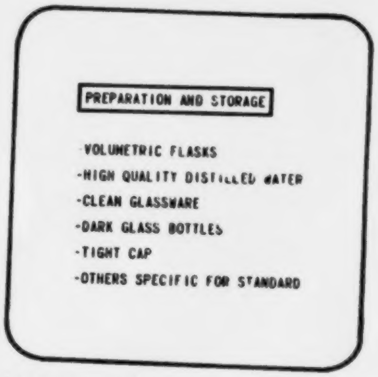
SLIDE 28



PRIMARY STANDARDS

1. FOR A BASE - POTASSIUM ACID PHTHALATE
   - SULFAMIC ACID
   - BENZOIC ACID
2. FOR AN ACID- SODIUM CARBONATE
   - CALCIUM CARBONATE
3. FOR SALTS - PURE FORM OF SUBSTANCE SALT WILL REACT WITH IN ANALYSIS.

SLIDE 29



PREPARATION AND STORAGE

-VOLUMETRIC FLASKS
-HIGH QUALITY DISTILLED WATER
-CLEAN GLASSWARE
-DARK GLASS BOTTLES
-TIGHT CAP
-OTHERS SPECIFIC FOR STANDARD

SLIDE 30

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: pH

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 45 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Participants need to know how to correctly standarize and use a pH meter to determine acidity and alkalinity, and as part of the analytical procedures for hardness, phosphorus, and nitrate and nitrite nitrogen.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of terms:

1. logarithm

2. acid

3. base

4. ion

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know the meaning of pH scale numbers, how to check pH equipment before use, how to standardize a pH meter using two buffer solutions and how to use the calibrated meter to determine pH.

B. Conditions: He/she will be given the training manual and 45 minutes of instruction. Later in the course, he/she will be given time and the necessary equipment to calibrate and use a pH meter.

C. Accepted Performance: Subsequent use of pH meter to correctly determine acidity and alkalinity in samples, according to an Instructor's rating. He/she must also correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the training manual, "Acidity, Alkalinity, pH and Buffers"

2. Seven overhead transparencies, OT-11:pH (See XI. Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the topic outline in the training manual and the visual materials. See XI below.

2. Prepare the lesson using Sequencing below or your own organization of the material.

3. Assemble meter, electrode(s), buffers, thermometer, beakers, distilled water, tissues, etc., to demonstrate use of a pH meter.

B. Sequencing:
Overheads are series OT-11:pH The training outline has reference information.

1. Overheads - The depth of this portion depends on the need and backgrounds of the participants. OT 4, 5, 6 and 7 topics may not be appropriate.

   a. Introduction - See IV above, Justification

   b. Basis and definition of pH scale-OT 1, 2

   c. Theory of operation utilized in pH meter-OT-3
   NOTE: A combination electrode has both functions.

   d. Construction and functions of electrodes-OT 4, 5, 6, 7
   NOTE: A combination electrode utilizes a specific metal chloride such as $AgCl_2$ along with the KCl.

2. Explanation with demonstration of:

   a. Checking a meter and electrode(s) for defects before use.

   b. Standardizing a meter using one buffer to set the meter and a second buffer to check for accurate read-out. Rinse between readings.
   NOTE: The first buffer should have a pH approaching that of the sample or of the end point desired in a titration procedure. The second buffer should have a pH about 4 units different from the first.

   c. Using the meter and electrode(s) to find pH values. Also rinsing between readings.

3. Note conditions for storing a meter and electrode(s).

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual materials

B. Equipment to demonstrate use of a pH meter.

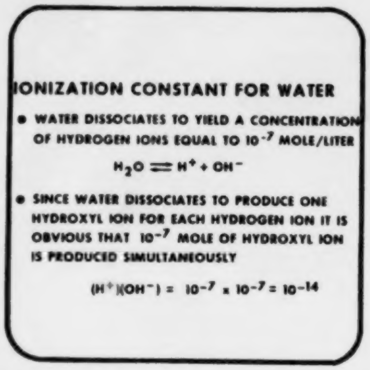
X. IPW REAGENT REQUIREMENTS:

A. None (Using a pH meter is utilized in subsequent laboratory assignments).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Seven overhead transparencies, OT-11:pH - See next page.

SERIES OT-11: pH



IONIZATION CONSTANT FOR WATER

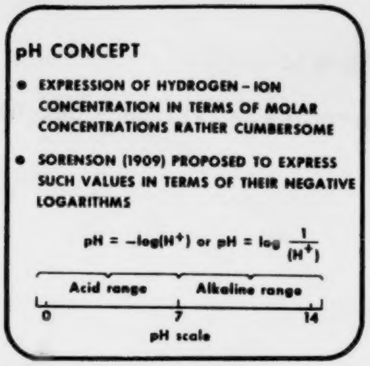
- WATER DISSOCIATES TO YIELD A CONCENTRATION OF HYDROGEN IONS EQUAL TO 10^{-7} MOLE/LITER

$$H_2O \rightleftharpoons H^+ + OH^-$$

- SINCE WATER DISSOCIATES TO PRODUCE ONE HYDROXYL ION FOR EACH HYDROGEN ION IT IS OBVIOUS THAT 10^{-7} MOLE OF HYDROXYL ION IS PRODUCED SIMULTANEOUSLY

$$(H^+)(OH^-) = 10^{-7} \times 10^{-7} = 10^{-14}$$

SLIDE 1



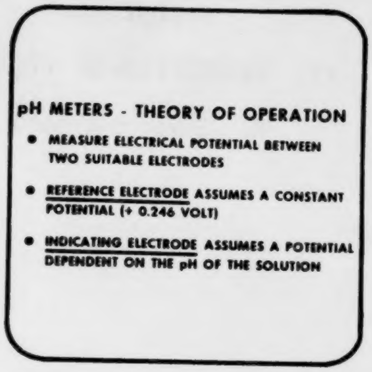
pH CONCEPT

- EXPRESSION OF HYDROGEN – ION CONCENTRATION IN TERMS OF MOLAR CONCENTRATIONS RATHER CUMBERSOME
- SORENSON (1909) PROPOSED TO EXPRESS SUCH VALUES IN TERMS OF THEIR NEGATIVE LOGARITHMS

$$pH = -\log(H^+) \text{ or } pH = \log \frac{1}{(H^+)}$$

Acid range
Alkaline range
0
7
14
pH scale

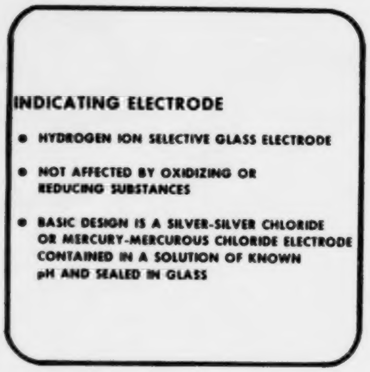
SLIDE 2



pH METERS - THEORY OF OPERATION

- MEASURE ELECTRICAL POTENTIAL BETWEEN TWO SUITABLE ELECTRODES
- REFERENCE ELECTRODE ASSUMES A CONSTANT POTENTIAL (+ 0.246 VOLT)
- INDICATING ELECTRODE ASSUMES A POTENTIAL DEPENDENT ON THE pH OF THE SOLUTION

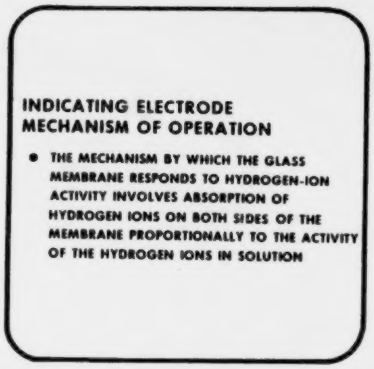
SLIDE 3



INDICATING ELECTRODE

- HYDROGEN ION SELECTIVE GLASS ELECTRODE
- NOT AFFECTED BY OXIDIZING OR REDUCING SUBSTANCES
- BASIC DESIGN IS A SILVER-SILVER CHLORIDE OR MERCURY-MERCUROUS CHLORIDE ELECTRODE CONTAINED IN A SOLUTION OF KNOWN pH AND SEALED IN GLASS

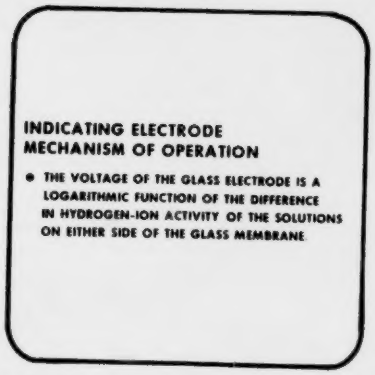
SLIDE 4



INDICATING ELECTRODE MECHANISM OF OPERATION

- THE MECHANISM BY WHICH THE GLASS MEMBRANE RESPONDS TO HYDROGEN-ION ACTIVITY INVOLVES ABSORPTION OF HYDROGEN IONS ON BOTH SIDES OF THE MEMBRANE PROPORTIONALLY TO THE ACTIVITY OF THE HYDROGEN IONS IN SOLUTION

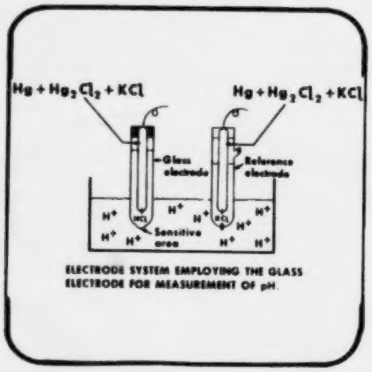
SLIDE 5



INDICATING ELECTRODE MECHANISM OF OPERATION

- THE VOLTAGE OF THE GLASS ELECTRODE IS A LOGARITHMIC FUNCTION OF THE DIFFERENCE IN HYDROGEN-ION ACTIVITY OF THE SOLUTIONS ON EITHER SIDE OF THE GLASS MEMBRANE.

SLIDE 6



ELECTRODE SYSTEM EMPLOYING THE GLASS ELECTRODE FOR MEASUREMENT OF pH.

SLIDE 7

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Acidity and Alkalinity

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 125 minutes (30+75+20)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

   A. Fundamental knowledge of inorganic chemistry

   B. Ability to correctly use volumetric glassware and titration assemblies

   C. Completion of the lessons (or equivalent):

      1. pH

      2. Volumetric Analysis

      3. Laboratory Safety Practices

   D. Ability to perform calculations involving decimals

VI. INSTRUCTIONAL OBJECTIVE:

   A. Terminal Behavior: The participant will know pertinent facts about acidity and alkalinity (see VIII B.) and will determine acidity in one sample and alkalinity in two samples using the procedures in the training manual (see VII A.1.c. and d.). He/she will calculate and report results and participate in a class discussion of the topics and laboratory procedures.

   B. Conditions: He/she will be given the training manual, 30 minutes of instruction, 75 minutes laboratory time, all necessary equipment, reagents and samples, and 20 minutes for class discussion.

   C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor, and obtain results for each sample within ± 10% of the class average (unless the reason for a lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

   A. Available Media:

      1. Four outlines in training manual:

         a. "Acidity, Alkalinity, pH and Buffers"

   b. "Alkalinity and Relationships Among the Various Types of Alkalinities"

   c. "Laboratory Procedure for Acidity" according to 1974 EPA Methods for Chemical Analysis, p. 1.

   d. "Laboratory Procedure for Total Alkalinity" according to 1974 EPA Methods for Chemical Analysis, p. 3

2. Twenty-eight slides, X-20: Alkalinity (See XI. Description of Visual Materials.)

B. Suggested Media: None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Prepare all necessary reagents and samples as in X below.

3. Review the laboratory procedures by performing the student assignment using the outlines in the training manual.

4. Review the topic outlines in the training manual and the visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Check pH meters for proper operation.

7. Distribute supplies and reagents to laboratory stations.

8. Assemble demonstration items: buret, pipet, pipet bulb and tissues.

B. Sequencing: Slides are series X-20: Alkalinity

1. Classroom Instruction on Alkalinity:

   a. Types of water samples of interest - Slide 1

   b. Definitions - Slides 2, 3

   c. Sources - Slides 4, 5

   d. Applications of data - Slides 6, 7, 8

   e. Sample handling - Slides 9, 10, 11, 12
   Note: Do not open bottle before anlysis (Slide 10)
   Note: Do not dilute samples (Slide 12)

   f. Procedure - Slides 13, 14, 15, 16, 17
   Note: 4.5 is the specified endpoint for NPDES data (Slide 13)
   Note: For alkalinity greater than 1000 mg, use 0.1N titrant. (Slide 15)

g. Calculations - Slides 18, 19
Recall: (N)(V) (eq. wt.) (1000/ml) which was developed in lesson on Volumetric Analysis

h. Chemistry involved in neutralizing the three major alkalinity sources. Stress that this is the reason for the 4.5 end point. (Slides 20, 21, 22, 23, 24, 25, 26)

2. Classroom Briefing on Alkalinity Laboratory - Use training manual outline, "Laboratory Procedure for Total Alkalinity."

   a. Calibrate pH meter. Set with pH 4.X buffer; check with pH 6.X buffer. (Briefly recall the steps which were demonstrated in the lesson on pH).

   b. Note section V with steps of alkalinity procedure. The acid has been standardized.

   c. When titrating to 4.5, record pH when a rapid change in pH occurs. Continue drop-by-drop titration to pH 4.5.

   d. Test two samples.

   e. Calculation formula is in procedure write-up.

3. Classroom Instruction on Acidity

   a. Sources - Strong mineral acids, weak acids, hydrolyzing salts

   b. Applications of data - Corrosivity of water; affects certain chemical and biological processes, can reflect changes in water quality.

   c. Sample handling - Same as for alkalinity

   d. Procedure Overview-Reason for 8.2 end point which represents neutralization of carbonic acid to the bicarbonate ion. Note: 8.2 is the specified end point for NPDES data.

4. Classroom Briefing on Acidity Laboratory-Use training manual outline, "Laboratory Procedure for Acidity".

   a. Section IV has steps of acidity procedure. The base has been standardized. For IV.D, caution about handling of hydrogen peroxide. Test one sample.

   b. Do alkalinity procedure while acidity sample is cooling.

   c. Calibrate pH meter. Set with pH 9.X buffer; check with pH 6.X buffer. (The steps involved were demonstrated in the lesson on pH).

   d. Calculation formula is in procedure write-up.

5. Summarize order of laboratory work.

6. Laboratory-Student performance of the assigned determinations (75 minutes)

7. Class Discussion (20 minutes)
   a. Board-Calculation formula reduced to factor of 20.
   b. Discuss significant figures to record for buret readings.
   c. Students calculate and post results. Also post ml of titrant used for each of the three samples.
   d. Find range of results.
   e. Announce theoretical results. See X. below.
   f. Demonstrate two corrections of technique error: removing air bubble in tip of buret and wiping excess off outside of pipet tip.
   g. Stress reason for 4.5 and 8.2 end points. Slide 27 has pH values associated with neutralizations. It can be used to discuss both endpoints. Stress that for acidity determinations for NPDES, the endpoint is 8.2, just below the 8.3 shown for bicarbonate ions in solution.
   h. Students report observations about rate of pH changes when titrating alkalinity samples.
   i. Relate observations to titration curves; rapid changes in pH during titration occur at the equivalence points shown by steeper curves on slide 28. Discuss applications shown in Table 1 of outline, "Alkalinity and Relationships Among the Various Types of Alkalinities". Slide 28 can be used to demonstrate the presence of contributors of alkalinity at the pH values used for the Table and which account for the titration results shown there.
   j. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assigment: 1 acidity sample and 2 alkalinity samples

A. For each station (no more than two persons per station):
   1. Three 100 ml beakers (for buffers)
   2. One 100 ml beaker (for titrants)
   3. One 150 ml beaker (for acid sample)
   4. One 150 ml beaker (for alkalinity samples)
   5. One 150 ml or larger beaker (for buret wastes)
   6. One large pipet bulb

7. One 25 ml buret mounted on a stand
8. One small funnel to fit the buret
9. One pH meter with electrode(s) in a 100 ml beaker with enough 6.X buffer or distilled water to cover the tip(s)
10. Magnetic stirring apparatus
11. One thermometer
12. Tongs (to transport 150 ml beaker)
13. One squeeze bottle of distilled water
14. One marking pencil
15. One apron for each student
16. One pair safety glasses for each student

B. Shared

1. X bottles for samples (3 samples, 1 of each per 3 stations)
2. X 50.0 ml volumetric pipets (one in each bottle of sample)
3. Two 50 ml beakers (in hood for H_2O_2)
4. Two medicine droppers (in hood for H_2O_2)
5. Supply of acid-cleaned glass boiling beads, 3 each acid sample (in hood)
6. Scoop to transfer boiling beads
7. Hot plates to accommodate the total number of 150 ml beakers containing acidity samples

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition, "Standard Methods", pages 276 and 279.

A. Minimum amounts per station (no more than two persons per station):

1. Seventy-five ml pH 4.X buffer
2. One hundred-fifty ml pH 6.X buffer
3. Seventy-five pH ml 9.X buffer
4. Five drops hydrogen peroxide, 30% solution
5. Twenty-five ml 0.020 N HCl or H_2SO_4 titrant
6. Fifteen ml 0.020 N NaOH titrant

7. Fifty ml acidity sample *(Dilute 300 ml 0.020 N HCl or H_2SO_4 to one liter. Fifty ml of this sample requires 15 ml 0.020 N NaOH to reach pH 8.2 and has 300 mg/L $CaCO_3$ acidity).

8. Fifty ml alkalinity sample *(Dilute 300 ml 0.020 N Na_2CO_3 solution to 1 liter. Fifty ml of this sample requires 15 ml 0.020 N HCl or H_2SO_4 to reach pH 4.5 and has 300 mg/L $CaCO_3$ alkalinity).

9. Fifty ml alkalinity sample *(Dilute 200 ml 0.020 N NaOH to 1 liter. Fifty ml of this sample requires 10 ml 0.020 N HCl or H_2SO_4 to reach pH 4.5 and has 200 mg/L $CaCO_3$ alkalinity).

*10. Wastewater treatment plant influents or effluents may be used as samples if they are known to contain acidity/alkalinity.

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory sessions.

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Twenty-eight slides, X-20: Alkalinity - See next pages.

SERIES X-20: ALKALINITY

ALKALINITY MEASUREMENTS

NATURAL WATERS

WASTEWATERS

DIGESTING SLUDGES

SLIDE 1

ALKALINITY

CAPACITY TO ACCEPT

HYDROGEN IONS (PROTONS)

SLIDE 2

ALKALINITY

CAPACITY TO

NEUTRALIZE ACIDS

SLIDE 3

HYDROXIDE $(OH)^-$

CARBONATE $(CO_3)^=$

BICARBONATE $(HCO_3)^-$

SLIDE 4

Salts of Weak Acids

acetates phosphates

borates

sulfides silicates

SLIDE 5

TREATMENT PLANTS

- AMENABILITY (to treatment)
- BUFFER CAPACITY
- ANAEROBIC DIGESTION
  COAGULATION
- INDUSTRIAL DISCHARGES

SLIDE 6

INDUSTRY

- BOILERS
- CORROSION CONTROL
- WATER SOFTENING

SLIDE 7

REGULATORY AGENCIES

- TREATED WATER
- ALKALINE DISCHARGES

SLIDE 8

PHOTO

OF

SAMPLE BOTTLES

SLIDE 9

$CO_2 \uparrow$

DO ALKALINITY
DETERMINATION
IMMEDIATELY

SLIDE 10

- 24 HOURS
- 4°C

SLIDE 11

DO
NOT
TREAT
SAMPLES

SLIDE 12

TITRATE
SAMPLES
TO
pH 4.5

SLIDE 13

PHOTO
OF
ADDING TITRANT

SLIDE 14

PHOTO
OF
TWO ACIDS

SLIDE 15

TITRATION

- RAPID
- MINIMAL AGITATION

SLIDE 16

APPROACHING pH 4.5

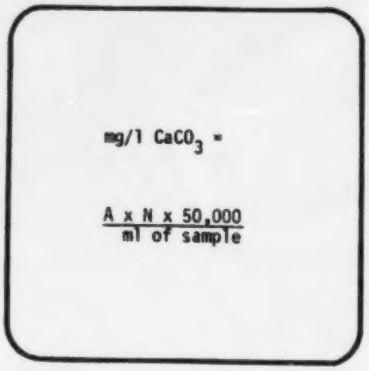
DROP-BY-DROP
ADDITIONS

SLIDE 17

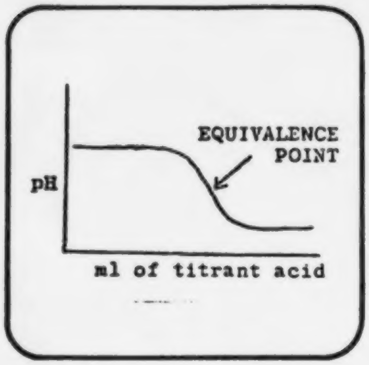
RESULTS

equivalent
calcium carbonate
milligrams/liter

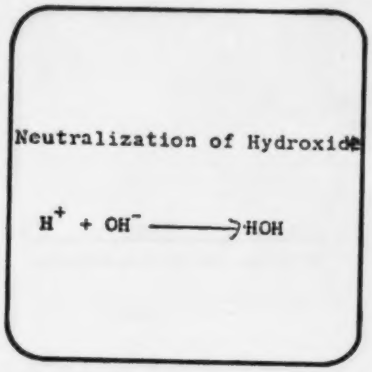
SLIDE 18


$$\text{mg/l } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml of sample}}$$

SLIDE 19

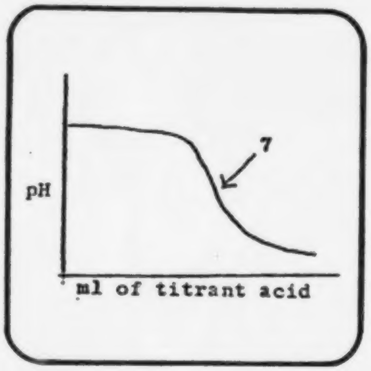


SLIDE 20

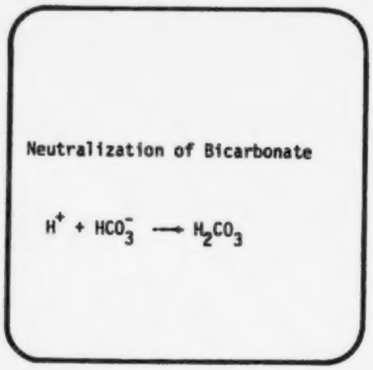

Neutralization of Hydroxide

$H^+ + OH^- \longrightarrow HOH$

SLIDE 21

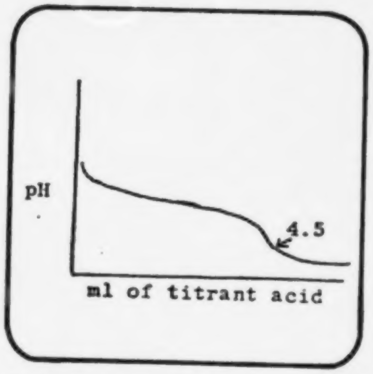


SLIDE 22

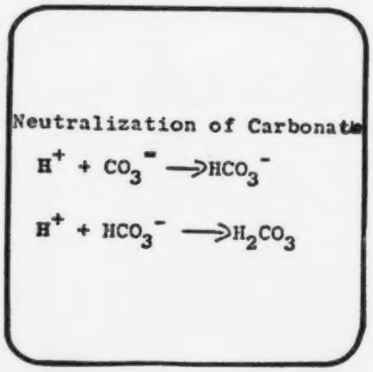

Neutralization of Bicarbonate

$H^+ + HCO_3^- \longrightarrow H_2CO_3$

SLIDE 23



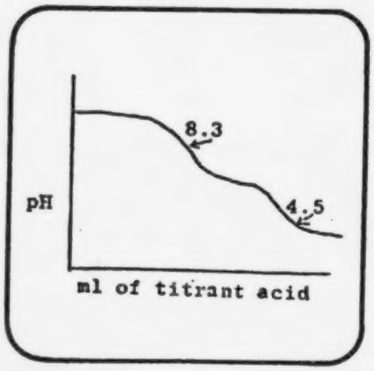
SLIDE 24


Neutralization of Carbonate

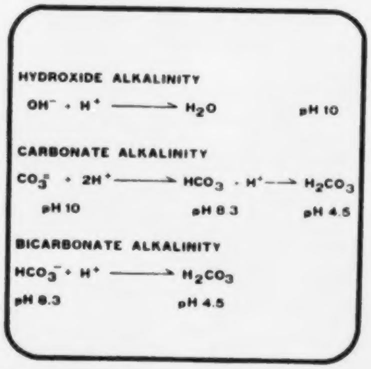
$H^+ + CO_3^= \longrightarrow HCO_3^-$

$H^+ + HCO_3^- \longrightarrow H_2CO_3$

SLIDE 25



SLIDE 26


HYDROXIDE ALKALINITY

$OH^- + H^+ \longrightarrow H_2O$ pH 10

CARBONATE ALKALINITY

$CO_3^= + 2H^+ \longrightarrow HCO_3 + H^+ \longrightarrow H_2CO_3$

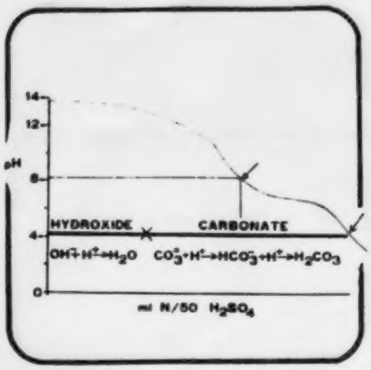
pH 10 pH 8.3 pH 4.5

BICARBONATE ALKALINITY

$HCO_3^- + H^+ \longrightarrow H_2CO_3$

pH 8.3 pH 4.5

SLIDE 27


14
12
pH
8
4
0
HYDROXIDE
CARBONATE
$OH^- + H^+ \rightarrow H_2O$
$CO_3^= + H^+ \rightarrow HCO_3^- + H^+ \rightarrow H_2CO_3$
ml N/50 H_2SO_4

SLIDE 28

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Indeterminate Error - Precision

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 60 minutes

IV. JUSTIFICIATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know how to check laboratory results for reproducibility (precision) to substantiate the reliability of his/her data.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of how to use formulas involving squares and square roots.

B. Though not essential, it is orderly to have completed the lessons (or equivalent):

1. Sample Handling

2. Compliance Methodology

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know the difference between accuracy and precision, the cause and synonyms for indeterminate error, the role of the normal distribution curve, where to find formulas and examples of each for variance, standard deviation, % relative standard deviation and range, how to establish one's precision in performing an analysis and how to continue to monitor precision on a daily basis Each will complete an assignment to calculate three precision statistics.

B. Conditions: He/she will be given the training manual and 60 minutes of instruction.

C. Accepted Performance: He/she must correctly identify at least 70% of the items pertaining to the objective in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in training manual:

a. "Statistics for Chemists"

b. "Accuracy - Precision - Error"

c. "Elements of a Quality Assurance Program"

2. Twenty slides, X-24: Precision (See XI Description of Visual Materials).

3. Five slides, X-26: Quality Assurance (See IPW on "Elements of a Quality Assurance Program," XI Description of Visual Materials).

4. HANDOUT: Assignment to calculate three statistics. A copy is at the end of this IPW.

5. HANDOUT: Key to assignment. A copy is at the end of this IPW.

6. Resource: EPA "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EMSL, Cincinnati, Ohio 45268.

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review visual materials and available handouts.

2. Prepare the lesson using the Sequencing below or your own organization of the material.

3. Duplicate copies of any handout materials.

B. Sequencing: Slides are X-24: Precision (Optional-slides X-26: Quality Assurance)

1. Introduction:

a. Before considering precision of results, one must have a valid sample and a skilled analyst using recognized analytical methodology.

b. Overview: Consideration of precision, i.e. the ability to reproduce results of chemical analyses, causes of error, and how to quantitate the effect of errors we cannot control. Statistics presented will be those most commonly used in recognized method references: variance for calculating standard deviation, % relative standard deviation, and range because it is an estimate of standard deviation.

c. Outlines: Formulas and examples of the calculations involved are in outline, "Statistics for Chemists" (S.C.). Discussion items from the outline "Accuracy - Precision - Error" are marked (A.P.E.).

2. Precision and Accuracy:

a. Analytical results are imperfect (A.P.E.) - Slide 1

b. Define/exemplify accuracy (A.P.E.) - Results are close to the true value-standard. Accuracy can also be checked by recovery of a constitutent - spike.- Slide 2.

c. Define/exemplify precision and contrast it to accuracy (A.P.E.) - Stress that both accuracy and precision are requirements for reliable data. - Slide 3.

3. Error

   a. Overview: Classification and causes of errors (A.P.E.) - Slide 4

   b. Synonyms for indeterminate error - Use (A.P.E.).

   c. Normal Distribution (S.C.) - Slide 5

4. Statistics

   a. Normal distribution is a population characteristic. An analyst deals with a sample of a population. Statistics are derived from samples. - Slide 6

   b. Define "statistic". - Slide 7

   c. Mean - Note location of aids to calculate in (S.C.) - Slide 8

   d. Variance and standard deviation - Slide 9

   e. Calculating variance - Slide 10, 11, 12

   f. Calculating standard deviation - Slide 13

      1) Note location of calculator formula (S.C.) - Slide 14
      Also note location of aids to manually calculate (S.C)

      2) Note variations of formula in (A.P.E.).

      3) Note EPA manual often uses standard deviation for precision statement.

   g. Relationship of standard deviation and normal distribution - Slide 15

   h. Normal distribution requirement and checking with probability paper - Slide 16

   i. Checking own precision (Elements of a Quality Assurance Program) - Slide 17

   j. Coefficient of variation (% relative standard deviation) (S.C.) - Slide 18 CONTINUED

1) Note concentration affects precision and accuracy.

2) Note Standard Methods often uses % relative standard deviation for precision statement.

k. Range - Slide 19

1) Note application in (A.P.E.) as estimate of standard deviation - Slide 20.

5. Daily Application

a. Suggested (EPA Handbook for AQC) - 10% of tests be duplicates to check precision

b. Can construct control charts with these statistics to monitor daily precision performance. (This topic is in Quality Assurance Program lesson. You may want to use Slides 1, 2, and 3 from X-26, Quality Assurance, now to demonstrate precision control charts).

6. Summary

a. Again stress valid sample and skilled analyst using recognized analytical methodology are prior to checking precision.

b. Summarize presentation.

7. Assignment

a. Hand out assignment to calculate three statistics outside of class time. Give a due time.

b. When due, collect the assignment and check the work of each to identify any questions.

c. Return assignment with a copy of the key for participant's reference.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual materials

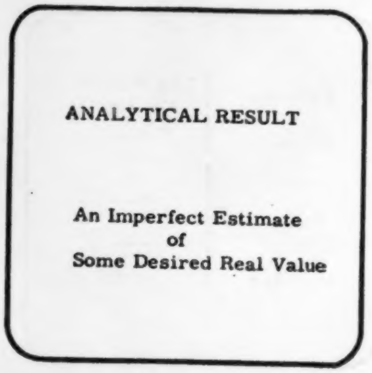
X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

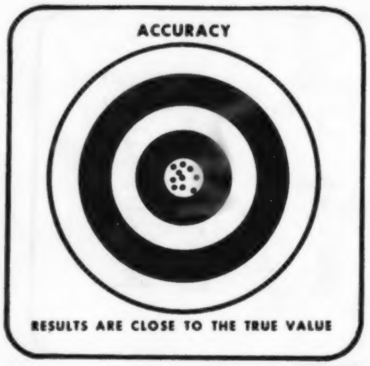
A. Twenty slides, X-24: Precision - see next pages

SERIES X-24: PRECISION


ANALYTICAL RESULT

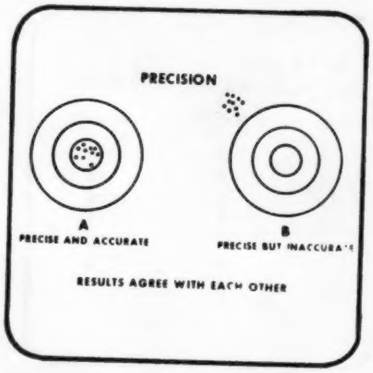
An Imperfect Estimate
of
Some Desired Real Value

SLIDE 1


ACCURACY

RESULTS ARE CLOSE TO THE TRUE VALUE

SLIDE 2

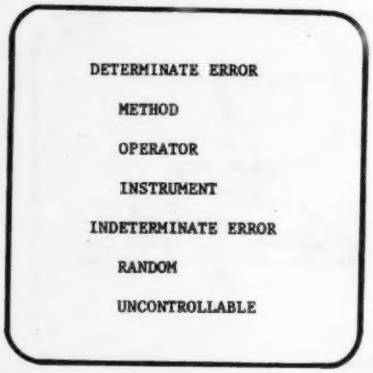

PRECISION

A
PRECISE AND ACCURATE

B
PRECISE BUT INACCURATE

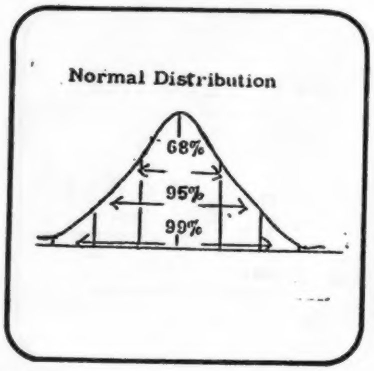
RESULTS AGREE WITH EACH OTHER

SLIDE 3

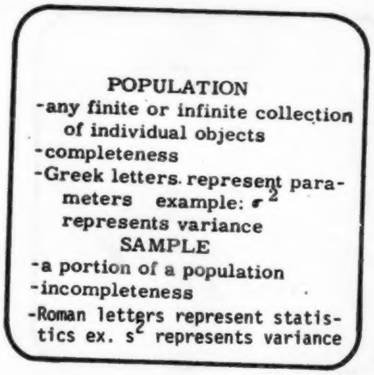

DETERMINATE ERROR
- METHOD
- OPERATOR
- INSTRUMENT

INDETERMINATE ERROR
- RANDOM
- UNCONTROLLABLE

SLIDE 4

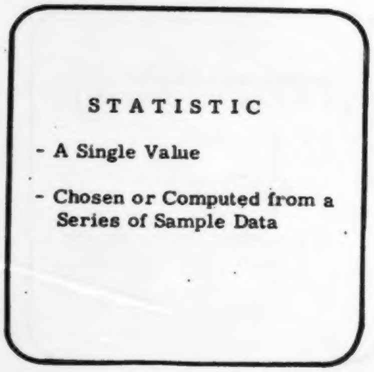

Normal Distribution

SLIDE 5

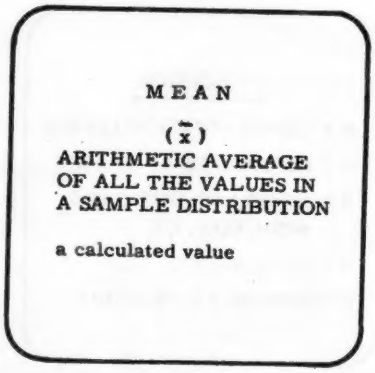

POPULATION
- any finite or infinite collection of individual objects
- completeness
- Greek letters represent parameters example: σ^2 represents variance

SAMPLE
- a portion of a population
- incompleteness
- Roman letters represent statistics ex. s^2 represents variance

SLIDE 6

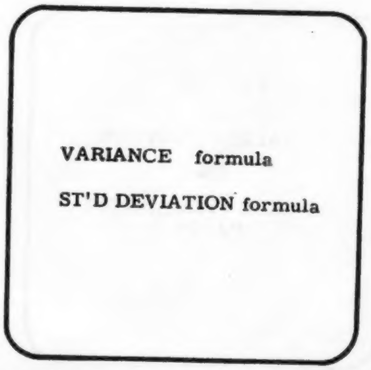

STATISTIC
- A Single Value
- Chosen or Computed from a Series of Sample Data

SLIDE 7


MEAN
$(\bar{x})$
ARITHMETIC AVERAGE OF ALL THE VALUES IN A SAMPLE DISTRIBUTION

a calculated value

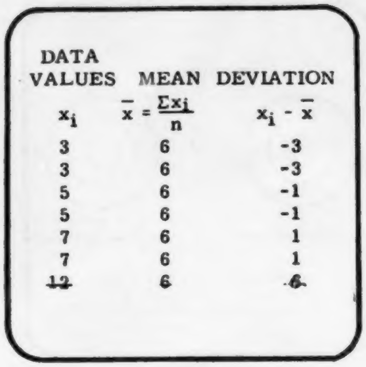
SLIDE 8


VARIANCE formula

ST'D DEVIATION formula

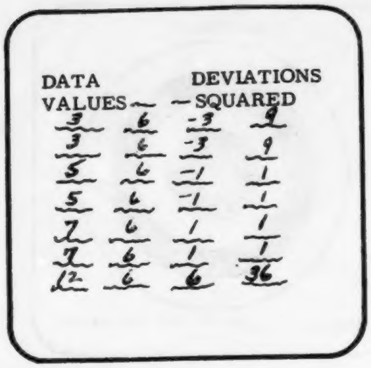
SLIDE 9

SERIES X-24: PRECISION



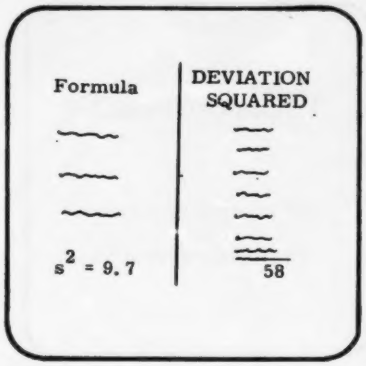
DATA VALUES x_i	MEAN $\bar{x} = \frac{\Sigma x_i}{n}$	DEVIATION $x_i - \bar{x}$
3	6	-3
3	6	-3
5	6	-1
5	6	-1
7	6	1
7	6	1
~~12~~	6	6

SLIDE 10



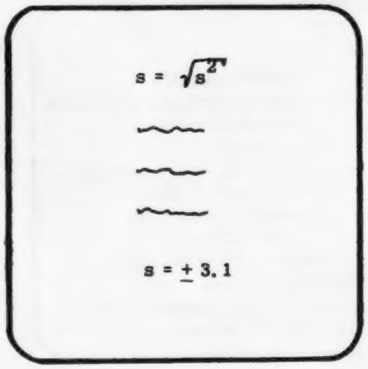
DATA VALUES			DEVIATIONS SQUARED
3	6	-3	9
3	6	-3	9
5	6	-1	1
5	6	-1	1
7	6	1	1
7	6	1	1
12	6	6	36

SLIDE 11



Formula	DEVIATION SQUARED
$s^2 = 9.7$	58

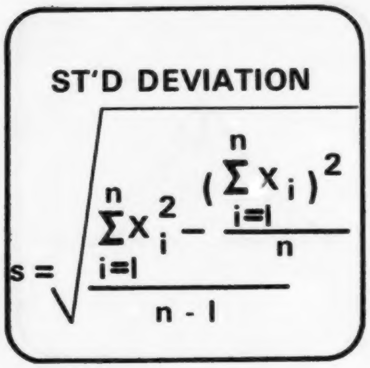
SLIDE 12



$s = \sqrt{s^2}$

$s = \pm 3.1$

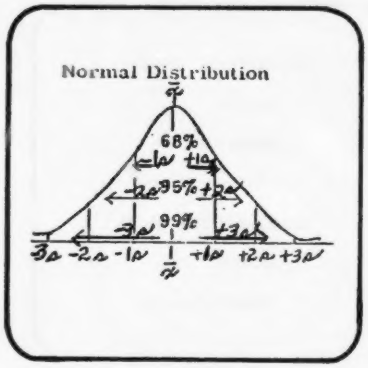
SLIDE 13



ST'D DEVIATION

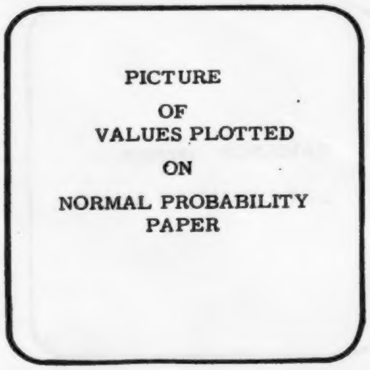
$$s = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \frac{\left(\sum_{i=1}^{n} x_i\right)^2}{n}}{n-1}}$$

SLIDE 14



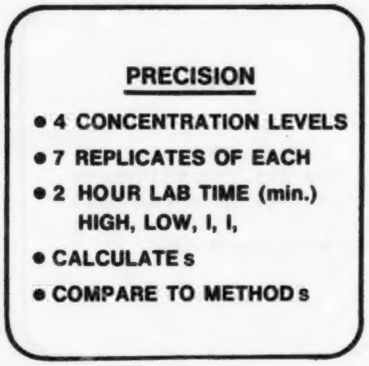
Normal Distribution

SLIDE 15



PICTURE
OF
VALUES PLOTTED
ON
NORMAL PROBABILITY
PAPER

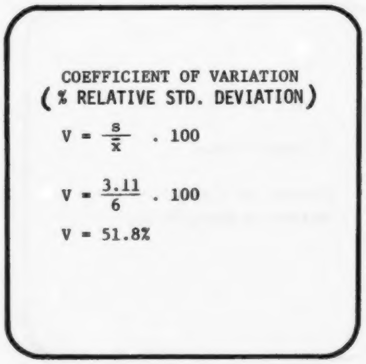
SLIDE 16



PRECISION

- 4 CONCENTRATION LEVELS
- 7 REPLICATES OF EACH
- 2 HOUR LAB TIME (min.)
  HIGH, LOW, I, I,
- CALCULATE s
- COMPARE TO METHOD s

SLIDE 17



COEFFICIENT OF VARIATION
(% RELATIVE STD. DEVIATION)

$V = \frac{s}{\bar{x}} \cdot 100$

$V = \frac{3.11}{6} \cdot 100$

$V = 51.8\%$

SLIDE 18

SERIES X-24: PRECISION

RANGE
(R)

MAXIMUM VALUE
MINUS
MINIMUM VALUE

SLIDE 19

R as ESTIMATE of s

$$\frac{R}{d_n} \sim s$$

SLIDE 20 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

PRECISION STATISTICS

NAME ______________________________

Given this set of **7** data items:

X_i
3
3
5
5
7
7
12

What is the standard deviation, S ? ______________________

$$S = \sqrt{\frac{\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 / n}{n-1}}$$

What is the % relative standard deviation, $\frac{S}{\overline{X}}100$? ______________________

What is the range, R ? ______________________________

PRECISION STATISTICS

Key

Given this set of 7 data items:

X_i	X_i^2
3	9
3	9
5	25
5	25
7	49
7	49
12	144
Σ 42	Σ 310

$$(\Sigma X_i)^2/n = 42^2/7$$

$$= 1764/7$$

$$= 252$$

What is the standard deviation, S ? ± 3.1

$$S = \sqrt{\frac{\sum_{i=1}^{n} X_i^2 - (\sum_{i=1}^{n} X_i)^2/n}{n-1}}$$

$$= \sqrt{\frac{310-252}{6}}$$

$$= \sqrt{\frac{58}{6}} = \pm 3.11$$

What is the % relative standard deviation, $\frac{S}{\overline{X}}100$? 52%

$$s = \pm 3.11$$

$$\overline{X} = \frac{42}{7} = 6.00$$

$$\frac{3.11}{6.00} \times 100 = 51.83\%$$

What is the range, R ? 9

$$12 - 3 = 9$$

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Calcium and Magnesium Hardness

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 92 minutes (40+45+7)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of inorganic chemistry

B. Ability to correctly use volumetric glassware and titration assemblies

C. Ability to perform calculations involving decimals

D. Completion of the lessons (or equivalent):

1. Volumetric Analysis

2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will learn pertinent facts about hardness (see VIII. B.) and will determine total hardness in a municipal wastewater treatment plant effluent using the procedure in the training manual (see VII. A. 1. b.). He/she will calculate and report results and participate in a class discussion of the topics and laboratory procedures.

B. Conditions: He/she will be given the training manual, 40 minutes of instruction, 45 minutes laboratory time, all necessary equipment and reagents, a sample, and 7 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor and obtain a result for the sample within ± 10% of the class average (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in training manual:

a. "Determination of Calcium and Magnesium Hardness"

b. "Laboratory Procedure for Total Hardness" according to 14th ed. Standard Methods, p 202

2. Twenty-three slides, X-22: Hardness (See XI. Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Prepare all necessary reagents and sample as in X below.

3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.

4. Review the topic outline in the training manual and the visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Distribute supplies and reagents to laboratory stations.

B. Sequencing: Slides are Series X-22: Hardness

1. Classroom Instruction - 40 minutes

   a. Name and units of the parameter as mentioned in the Federal Register - Slide 1.

   b. Partial definition of hardness, excerpted from old US PHS literature - Slide 2.

   c. This slide is an extension of Slide 2 - Slide 3.

   d. Other hardness - causing cations - Slide 4.

   e. Anions associated with the hardness - causing cations - Slide 5.

   f. Reasons why waters differ in degrees of hardness - Slide 6.

   g. Ranges of hardness, according to Sawyer - Slide 7.

   h. Processes for removing hardness - Slide 8.

   i. Ionization/dissociation of inorganic and organic acids - Slides 9 and 10. The purpose of these slides is to show, by analogy to simple inorganic and organic acid molecules, how a soap molecule is formed via neutralization.

   j. How calcium forms an insoluble precipitate with a soap molecule - Slide 11.

   k. Federal Register methods for determining surfactants - Slide 12.

l. Application and range of the EDTA titration method, as outlined in the 1974 EPA Methods Manual - Slide 13.

m. Steps in the EDTA titration method - Slide 14.

The metal in step 1 is that which is part of the indicator; that in step 2 is what is in the sample.

n. Indicators used in the EDTA titration procedure - Slide 15.

o. Two dimensional representation of the Calcium - EDTA complex - Slide 16.

p. Problems in the EDTA titration procedure - Slide 17.

q. Interferences in the EDTA procedure - Slide 18.

r. Two forms of calcium and magnesium which may be determined - Slide 19.

s. Indicators used in the EDTA titration of calcium - Slide 20.

t. Non-Federal Register methods for determining magnesium - Slide 21 and 22.

u. Non-Federal Register method for determining hardness - Slide 23.

2. Laboratory - 45 minutes - Use training manual

a. Briefing in laboratory

1) Calibrate pH meter. Set with pH 9.X buffer; check with pH 6.X buffer.

2) Do Section II of the laboratory outline using 25 ml tap water instead of the 25 ml of $CaCO_3$ indicated in II.A. This allows participants to become accustomed to the color changes.

3) Repeat Section II, this time using $CaCO_3$.

4) Calculate the "B" factor according to Section IV.A.

5) Titrate the sample according to Section III.

6) Calculate total hardness for the sample according to Section IV.B.

b. Student performance of the assignment

3. Class Discussion - 7 minutes
    a. Students post results.
    b. Discuss results, exclude outliers, calculate the average.
    c. Cover any items from laboratory session.
    d. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Three titrations - tap water, $CaCO_3$ standard solution, municipal wastewater treatment plant sample

A. For each station (no more than two persons per station):

1. One 100 or 150 ml beaker (for buffer)
2. One small beaker (for buret wastes)
3. One 25 ml buret mounted on a stand
4. One small funnel to fit the buret
5. One 10 ml graduated cylinder
6. One 25 ml graduated cylinder
7. One 25 ml volumetric pipet
8. One pipet bulb
9. One small spatula or measuring scoop (for 0.2 g of indicator)
10. One pH meter with electrode(s) in a beaker with enough 6.X buffer or distilled water to cover the tip(s). Also, one thermometer and stirring mechanism.
11. One flask, 125 ml Erlenmeyer if a combination pH electrode is used. Otherwise, a 150 ml beaker.
12. One squeeze bottle of distilled water
13. One marking pencil
14. One apron for each student
15. One pair of safety glasses for each student

B. Shared:

1. None

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition, "Standard Methods", page 203.

A. Minimum amounts per station (no more than two persons per station):

1. Twenty-five ml calcium carbonate standard, 1 mg $CaCO_3$/ml

2. Six ml buffer solution (in a pipettor; otherwise; use dropper bottles or a graduated pipet)

3. Seventy-five ml disodium ethylenediamine tetraacetic acid dihydrate

4. One hundred ml pH 6.X buffer

5. One hundred ml pH 9.X buffer

6. Six tenths g indicator, Eriochrome Black T. (See Standard Methods for commercial equivalents).

7. Twenty-five one hundredths g inhibitor. This is usually not required. (See Standard Methods for discussion and choices).

8. Twenty-five ml municipal wastewater treatment plant effluent for sample (If not available, use a calcium carbonate solution with concentration withing 15 ml titrant volume limit).

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Twenty-three slides, X-22, Hardness - See next pages.

SERIES X-22: HARDNESS

TOTAL HARDNESS

mg $CaCO_3$/l

SLIDE 1

HARDNESS DEFINITION

...the total concentration of just the calcium and magnesium ions.....

SLIDE 2

HARD WATER - HIGH IN CALCIUM, MAGNESIUM, AND IRON IONS RELATIVE TO THE AMOUNT OF SODIUM AND POTASSIUM IONS.

SOFT WATER - LOW IN CALCIUM, ...

SLIDE 3

OTHER CONTRIBUTORS TO HARDNESS

- **ALUMINUM**
- **IRON**
- **MANGANESE**
- **STRONTIUM**
- **HYDROGEN IONS**

SLIDE 4

ASSOCIATED ANIONS

- HCO_3^-
- SO_4^{-2}
- Cl^-
- NO_3^-
- SiO_4^{-2}

SLIDE 5

VARIATIONS IN HARDNESS

- **LEACHING OF DIFFERENT SOIL AND ROCK**
- **WASTE DISCHARGE**
- **USAGE OF THE WATER**
  - → **IRRIGATION**
  - → **SOFTENING**

SLIDE 6

HARDNESS CLASSIFICATION

	MG/L
SOFT	0-75
MODERATELY HARD	75-150
HARD	150-300
VERY HARD	>300

SLIDE 7

HARDNESS REMOVAL

- **ZEOLITES**
- **LIME SODA**
- **HOT PHOSPHATE**
- **COMPLEX FORMATION**
- **DISTILLATION**

SLIDE 8

Inorganic (Mineral) Acid

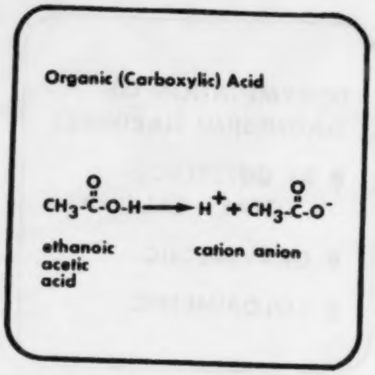
$$HCl \longrightarrow H^+ + Cl^-$$

cation hydrogen ion proton

anion chloride ion

SLIDE 9

SERIES X-22: HARDNESS

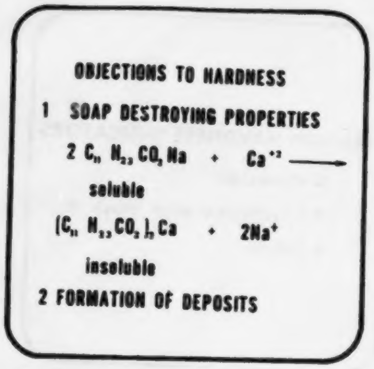

Organic (Carboxylic) Acid

$$CH_3\text{-}\overset{O}{\overset{\|}{C}}\text{-}O\text{-}H \longrightarrow H^+ + CH_3\text{-}\overset{O}{\overset{\|}{C}}\text{-}O^-$$

ethanoic acetic acid

cation anion

SLIDE 10


OBJECTIONS TO HARDNESS

1 SOAP DESTROYING PROPERTIES

$2\ C_{11}H_{23}CO_2Na + Ca^{+2} \longrightarrow$

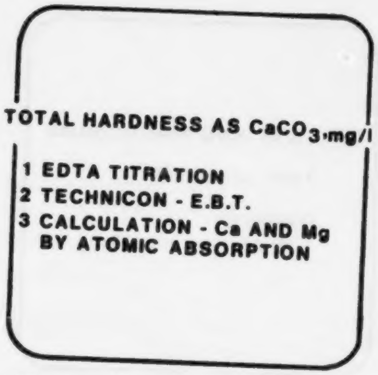
soluble

$(C_{11}H_{23}CO_2)_2Ca + 2Na^+$

insoluble

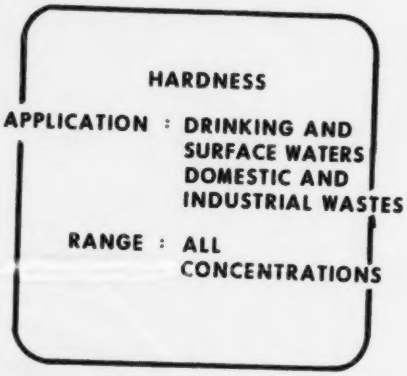
2 FORMATION OF DEPOSITS

SLIDE 11


TOTAL HARDNESS AS $CaCO_3$-mg/l

1 EDTA TITRATION
2 TECHNICON - E.B.T.
3 CALCULATION - Ca AND Mg BY ATOMIC ABSORPTION

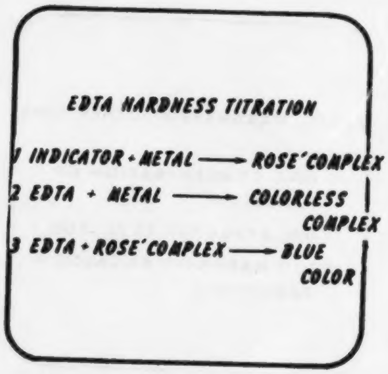
SLIDE 12


HARDNESS

APPLICATION : DRINKING AND SURFACE WATERS DOMESTIC AND INDUSTRIAL WASTES

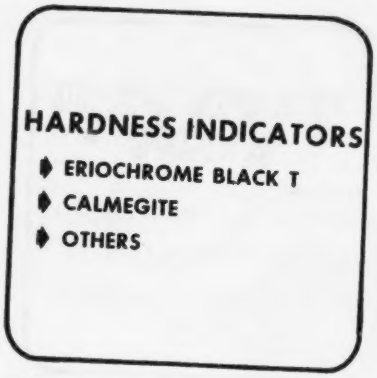
RANGE : ALL CONCENTRATIONS

SLIDE 13


EDTA HARDNESS TITRATION

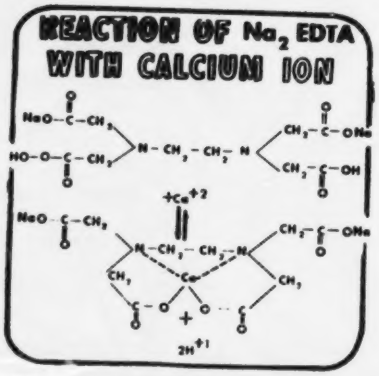
1 INDICATOR + METAL ⟶ ROSE' COMPLEX
2 EDTA + METAL ⟶ COLORLESS COMPLEX
3 EDTA + ROSE' COMPLEX ⟶ BLUE COLOR

SLIDE 14

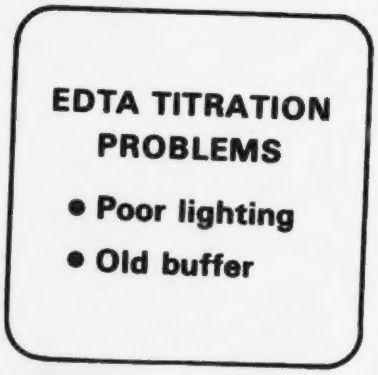

HARDNESS INDICATORS

- ERIOCHROME BLACK T
- CALMEGITE
- OTHERS

SLIDE 15

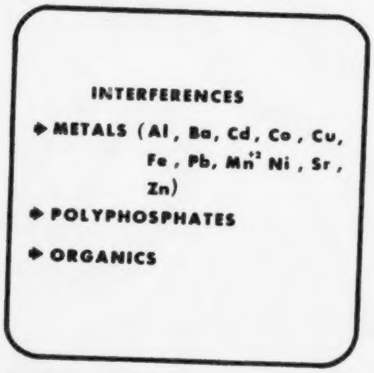

REACTION OF Na_2 EDTA WITH CALCIUM ION

SLIDE 16


EDTA TITRATION PROBLEMS

- Poor lighting
- Old buffer

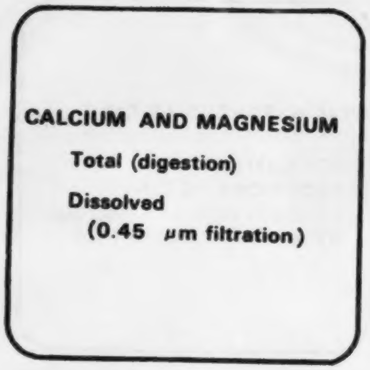
SLIDE 17


INTERFERENCES

- METALS (Al, Ba, Cd, Co, Cu, Fe, Pb, Mn^{+2} Ni, Sr, Zn)
- POLYPHOSPHATES
- ORGANICS

SLIDE 18

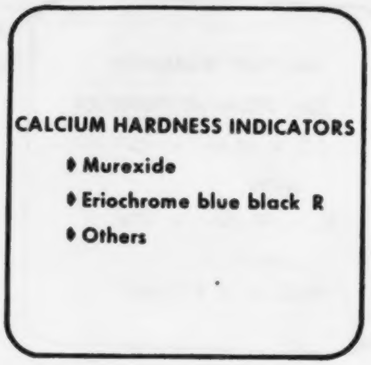
SERIES X-22: HARDNESS


CALCIUM AND MAGNESIUM

Total (digestion)

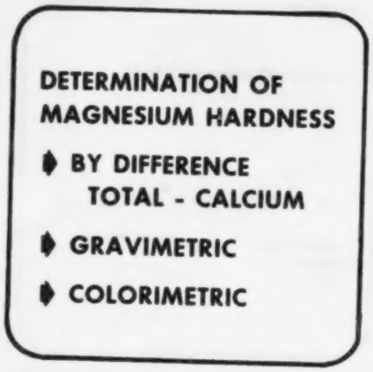
Dissolved
(0.45 μm filtration)

SLIDE 19


CALCIUM HARDNESS INDICATORS

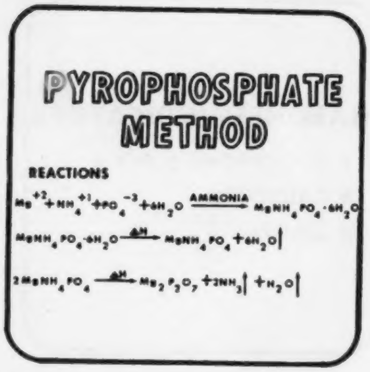
- Murexide
- Eriochrome blue black R
- Others

SLIDE 20


DETERMINATION OF MAGNESIUM HARDNESS

- BY DIFFERENCE
  TOTAL - CALCIUM
- GRAVIMETRIC
- COLORIMETRIC

SLIDE 21


PYROPHOSPHATE METHOD

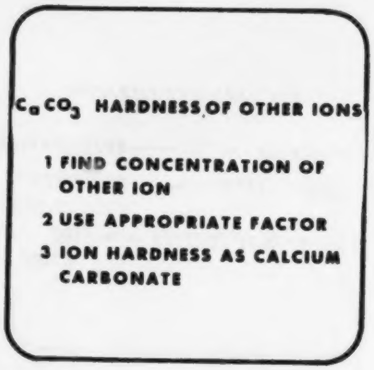
REACTIONS

$$Mg^{+2} + NH_4^{+1} + PO_4^{-3} + 6H_2O \xrightarrow{\text{AMMONIA}} MgNH_4PO_4 \cdot 6H_2O$$

$$MgNH_4PO_4 \cdot 6H_2O \xrightarrow{\Delta H} MgNH_4PO_4 + 6H_2O\uparrow$$

$$2MgNH_4PO_4 \xrightarrow{\Delta H} Mg_2P_2O_7 + 2NH_3\uparrow + H_2O\uparrow$$

SLIDE 22


$CaCO_3$ HARDNESS OF OTHER IONS

1. FIND CONCENTRATION OF OTHER ION
2. USE APPROPRIATE FACTOR
3. ION HARDNESS AS CALCIUM CARBONATE

SLIDE 23

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Chlorination and Chlorine Determinations

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 98 minutes (45 + 45 + 8)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

- A. Fundamental knowledge of inorganic chemistry
- B. Ability to correctly use volumetric glassware and titration assemblies
- C. Ability to perform calculations involving decimals
- D. Completion of the lessons (or equivalent):
  - 1. Volumetric Analysis
  - 2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will learn about chlorination products and terminology, and the chemistry involved in chlorine determinations by iodometric analyses and DPD methodology. He/she will twice determine total chlorine residual in a sample by back titration using amperometric apparatus (See VII A.1.b.), calculate and report results from the second determination and participate in a class discussion of the results.

B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 45 minutes laboratory time and all necessary equipment, and 8 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor and obtain a result for the sample within ± 10% of the class average (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in training manual:

- a. "Chlorination and Chlorine Determinations"
- b. "Amperometric Determination of Total Residual Chlorine" according to 14th edition Standard Methods, page 318 for reagent preparation and Instruction Manual, Amperometric Titrator (Wallace and Tiernan) for procedure.

2. Thirty-six slides, X-21: Chlorine (See XI. Description of Visual Materials).

3. OPTIONAL: KEH-70: 11 minute CCTV cassette on "Amperometric Determination of Total Chlorine in Wastewater" presents the stepwise procedure for the back titration using a Wallace and Tiernan titrator. Produced by Kirkwood Community College, it is available for review and purchase (6301 Kirkwood Blvd., Cedar Rapids, Iowa 52406).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.
2. Prepare all necessary reagents and sample as in X below.
3. Review the laboratory procedure by perfoming the student assignment using the outline in the training manual.
4. Review the topic outline in the training manual and the visual materials. See XI below. If you plan to use VC-116 rather than demonstrate the laboratory procedure, order and review the tape.
5. Prepare the lesson using the Sequencing below or your own organization of the material.
6. Check amperometric titrators for accurate operation. Clean and condition cells if necessary.
7. Distribute supplies and reagents to laboratory stations.
8. If you plan to demonstrate the back titration procedure with an amperometric titrator and/or starch-iodide titration apparatus, assemble the equipment and reagents.
9. If you plan to demonstrate the DPD titration and/or spectrophotometer method(s), assemble the equipment and reagents.

B. Sequencing:

Slides are Series X-21: Chlorine

Participants should use the training outline, "Chlorination and Chlorine Determinations" as a reference for this information.

1. Classroom instruction on chlorination, using slides to discuss the first 3 Sections of the outline in the training manual.

   a. Introduction about bactericidal use of chlorine - Slide 1

   b. Means of chlorination - Slide 2

c. Effects of applying chlorine gas - Slide 3, 4

d. Effects of applying calcium hypochlorite - Slide 5, 6

e. Ammonia reactions with hypochlorous acid produce chloramines - Slide 7, 8

f. Side reactions reduce availability of chlorine for disinfecting purposes - Slide 9

g. Chlorination products that have disinfection powers - Slide 10

h. Stress the meaning of the terms related to chlorine residuals - Slide 11, 12, 13

i. Factors affecting disinfection - Slide 14

j. It is convenient to use a blank slide (15) here.

2. Classroom overview of recognized chlorine determinations, using headings in training manual sections on "Iodometric Analyses" and "DPD Methodology", then the summary of "Compliance Methodology".

3. Classroom instruction on the chemistry involved in direct iodometric titrations to determine free, combined or total chlorine. This series of slides presents facts about the chemistry of the method with pictures of an analyst doing each step using an amperometric titrator. Presentation of the direct method gives an opportunity to teach the chemistry involved and also to re-enforce mastery of the meaning of the terms "free," "combined", and "total" residual chlorine. However, for wastewaters the indirect or back titration is to be used. If participants will routinely analyze wastewater, you can still use these slides to teach the chemistry involved and the meaning of the above terms, but spend more time on the actual steps to do a back titration (next topic).

   A summary of Iodometric Titration Analyses is in Section IV of the outline in the training manual. Participants can use this as a reference for the following information:

   a. Types of chlorine - Slide 16

   b. Role of phenylarsine oxide - Slide 17

   c. Picture of titration equipment - Slide 18

   d. pH for types - Slide 19

   e. Principle for free chlorine - Slide 20

   f. 1 ml pH 7 buffer has been added and current flows, pointer to right - Slide 21

g. PAO reduces, current reduced - Slide 22

h. Adjust pointer to right - Slide 23

i. More PAO, current reduced - Slide 24

j. Needle stops, take reading - Slide 25

k. More PAO. When needle stays stopped, it's end. (Slide 26 is same as slide 25 to show needle staying same) - Slide 26

l. 0.00564 N PAO related to chlorine for a 200 ml sample - Slide 27

m. PAO related to free chlorine at pH 6.5 - 7.5 - Slide 28

n. Combined chlorine may be present - Slide 29

o. Utilize chlorine - iodide reaction - Slide 30

p. Need pH 4 and potassium iodide - Slide 31

q. Combined chlorine releases iodine - Slide 32

r. Add KI solution (crystals may be used instead of a solution) - Slide 33

s. Add pH 4 buffer - Slide 34

t. Repeat titration procedure as for free chlorine; when needle stays at one place, it is the end point - Slide 35

u. Add results for free and for combined chlorine to give total. Stress: can do total in one step by adding KI and pH 4 buffer directly to sample - Slide 36

4. Classroom instruction/demonstration on indirect (back titration) iodometric procedures to determine total chlorine.Reference information is in the outline in the training manual, Section IV.

   a. The indirect or back titration is required for wastewaters because constituents in the sample can react with liberated iodine during titration and cause low results for the PAO reaction with liberated iodine.

   b. Utilize same principles as the direct method. Stress additional step using iodine titrant and that the end point signal is reversed.

   c. CHOICE of instructional approach:

      1) Eleven minute CCTV cassette showing the back titration using an amperometric titrator.

      2) Demonstrate a back titration using an amperometric titrator and/or the starch-iodide colorimetric procedure. The latter requires addition of starch for end point detection.

5. Classroom instruction on the DPD Method to determine free or total chlorine. Section V of the outline in the training manual has the following information:

   a. Chemistry involved is similar to iodometric method, depending on liberation of iodine from potassium iodide.

   b. Role of DPD to produce color.

   c. Ferrous ammonium sulfate is used as titrant. Note end point signal.

   d. Spectrophotometric method

   e. OPTIONAL: Demonstrate one or both DPD methods.

6. A summary of methodology recognized for compliance monitoring is in Section VI

7. In laboratory - 45 minutes - Briefing which includes equipment preparation information and instrument controls, then student performance of two determinations of total chlorine in a sample by back titration using amperometric apparatus. Use training manual outline, "Amperometric Determination of Total Residual Chlorine."

8. Class Discussion - 8 minutes

   a. Students calculate and post results.

   b. Announce result obtained by Instructor.

   c. Discuss problems responsible for wrong results.

   d. Note sample collection and analysis cautions.

   e. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 total chlorine sample. Run one determination for practice, run second determination to report results.

To minimize the number of titrators required, the laboratory session could be conducted twice with half the class doing another procedure requiring about the same time, e.g., Hardness.

A. For each station (no more than two persons per station):

   1. One 1 ml pipet graduated in tenths
   2. One 5 ml volumetric pipet (Use a graduated pipet if using the delivery assembly for PAO on a titrator).
   3. One 200 ml graduated cylinder
   4. One propipet bulb

5. One scoop for KI crystals
6. One box tissues
7. One container for waste solutions
8. One amperometric titrator with cell containing electrolyte and conditioned for use
9. One squeeze bottle chlorine - free distilled water
10. One marking pencil
11. One apron for each student
12. One pair safety glasses for each student

B. Shared:

1. X bottles for sample (1 per 2 stations)

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition "Standard Methods", page 318.

A. Minimum amounts per station (no more than two persons per station):

1. Ten ml 0.00564N phenylarsine oxide solution
2. Two g potassium iodide crystals (or one dropper full of KI solution)
3. Eight ml pH 4 acetate buffer (in dropper bottle)
4. Two ml 0.0282N iodine titrant (in dark bottle)
5. Four hundred ml sample with total chlorine residual around 3mg/L (A chlorine solution may be diluted. Another source is a hypochlorite bleach or a chlorinated wastewater treatment plant effluent may be used. Check any solution to be used as a sample to be sure chlorine is present in the applicable range of the test).

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of chlorine-free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Thirty-six slides, X-21: Chlorine - See next pages.

SERIES X-21: CHLORINE

Cartoon-type of drawing showing plant operator killing disease organisms with chlorine.

SLIDE 1

.Chlorine gas

.Chlorine gas dissolved in water

.Calcium hypochlorite

SLIDE 2

.Chlorine gas

.Chlorine gas dissolved in water
↓
pH Decrease

SLIDE 3

PRODUCTS of Cl_2

H^+ - hydrogen ion
Cl^- - chloride ion
HOCl - hypochlorous acid
⇅ H^+ - hydrogen ion
OCl^- - hypochlorite ion

SLIDE 4

.Calcium hypochlorite
↓
pH Increase

SLIDE 5

PRODUCTS of $Ca(OCl)_2$

Ca^{+2} - calcium ion
OH^- - hydroxide ion
HOCl - hypochlorous acid
⇅ H^+ - hydrogen ion
OCl^- - hypochlorite ion

SLIDE 6

Ammonia

SLIDE 7

.Monochloramines

.Dichloramines

.Trichloramines

SLIDE 8

Demand from side reactions

SLIDE 9

SERIES X-21: CHLORINE

Disinfection

.Monochloramines

.Dichloramines

.Hypochlorite ion

SLIDE 10

Free Chlorine residuals

.Chlorine

.Hypochlorous acid

.Hypochlorite ion

SLIDE 11

Combined Chlorine

.Chloramines

SLIDE 12

Total residual chlorine = free chlorine residual + combined chlorine residual

SLIDE 13

Disinfecting power

.Concentration

.Contact time

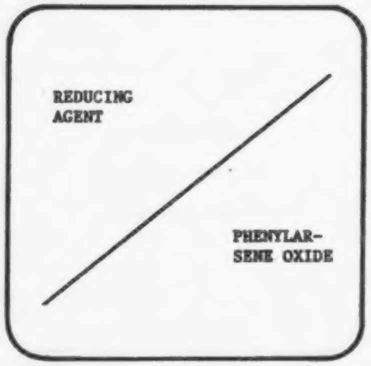
SLIDE 14

BLANK SPACER SLIDE

SLIDE 15

FREE CHLORINE - MG/1
+
COMBINED CHLORINE - MG/1
↓
TOTAL RESIDUAL CHLORINE

SLIDE 16


REDUCING AGENT

PHENYLARSENE OXIDE

SLIDE 17

LIVE OF TITRATION ASSEMBLY

SLIDE 18

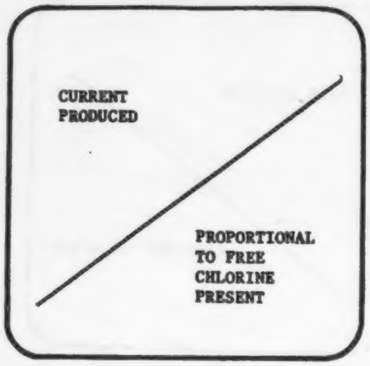
SERIES X-21: CHLORINE

TWO STAGE TITRATION

pH 7 FREE CHLORINE RESIDUAL

pH 4 COMBINED CHLORINE RESIDUAL

SLIDE 19



CURRENT PRODUCED

PROPORTIONAL TO FREE CHLORINE PRESENT

SLIDE 20

LIVE OF ADDING TITRANT,

POINTER TO RIGHT

SLIDE 21

LIVE OF ADDING TITRANT,

CURRENT REDUCED

SLIDE 22

LIVE OF ADJUSTING POINTER

TO RIGHT ON SCALE

SLIDE 23

LIVE OF ADDING MORE TITRANT

SLIDE 24

LIVE OF POINTER AT REST

SLIDE 25

LIVE OF POINTER STAYING AT REST

SLIDE 26

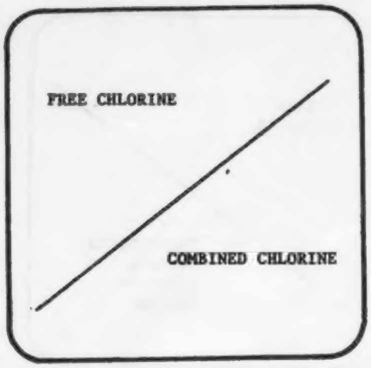
1 ml PHENYLARSENE OXIDE
EQUALS 1 mg/l OF CHLORINE

SLIDE 27

SERIES X-21: CHLORINE

PHENYLARSENE OXIDE REACTS
WITH FREE CHLORINE ONLY

SLIDE 28



SLIDE 29

STARCH-IODIDE METHOD

CHLORINE

OXIDIZES

IODIDE TO PRODUCE FREE

→IODINE

SLIDE 30

COMBINED CHLORINE DETERMINATION
pH 4 AND ADDITION
OF POTASSIUM IODIDE

SLIDE 31

COMBINED CHLORINE COMPOUNDS

OXIDIZE IODIDE
↓
FREE IODINE

SLIDE 32

LIVE OF ADDING KI SOLUTION

SLIDE 33

LIVE OF ADDING pH4 BUFFER

SLIDE 34

LIVE OF REPEATING TITRATION

PROCESS

SLIDE 35

FREE CHLORINE - MG/1
+
COMBINED CHLORINE - MG/1
↓
TOTAL RESIDUAL CHLORINE

SLIDE 36 (END OF SLIDES)

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Use of a Spectrophotometer and Calibation Graphs

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 45 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Participants need to use a spectrophotometer and prepare calibration graphs to determine total phosphorus, nitrate and nitrite nitrogen, and fluoride.

V. ENTRY LEVEL BEHAVIOR:

A. Ability to perform calculations involving decimals

B. Knowledge of weight and volume terms like "mg", "μg", "L", "ml", "standard solution"

C. Ability to use arithmetic graph paper

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know how to zero a spectrophotometer at a selected wavelength, make absorbance measurements for standards and samples, use the results to prepare a calibration graph and then use the graph to determine the concentration of the desired constituent in a sample, all according to the outline in the training manual (see VII. A. 1.).

B. Conditions: He/she will be given the training manual and 45 minutes of instruction. Later in the course, he/she will be given the time and the necessary equipment to perform spectrophotometric measurements and to prepare calibration graphs.

C. Accepted Performance: Subsequent satisfactory use of a spectrophotometer and ability to prepare calibration graphs for the total phosphorus and the nitrate and nitrite nitrogen determinations, all according to an Instructor's rating.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in training manual: "Use of a Spectrophotometer". Terms and operational sequence are for B & L Spectronic 20 models, but can be easily adapted for any spectrophotometer.

2. Sixteen slides, X-8: Use of a Spectrophotometer (see XI Description of Visual Materials).

3. Six slides, X-9: Calibration Graphs (see XI Description of Visual Materials).

4. Thirty-five slides, 8 minute tape and script, XT-51: Use of the

Spectronic 20 Spectrophotometer (See XI Description ov Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the course laboratory procedures which utilize a spectrophotometer: phosphorus, fluoride, nitrate and nitrite nitrogen.
2. Review visual materials and outline in course manual.
3. Prepare the lesson using the Sequencing below or your own organization of the material.

B. Sequencing:

Slides are series X-8: Use of a Spectrophotometer and X-9: Calibration Graphs. Slide-tape presentation is XT-51: Use of the Spectronic 20 Spectrophotometer.

1. Classroom - Discuss colorimetry, Lambert-Beer Law, and preparation of standards using X-8; Use of a Spectrophotometer.
    a. General principles of absorbance and colorimetry; X-8, 1.
    b. Definition of wavelength; X-8, 2.
    c. Characteristics of wavelength; X-8, 3.
    d. Relation of wavelength values to inches; X-8, 4.
    e. Lambert-Beer Law; X-8, 5.
    f. Shapes of absorption cells; X-8, 6.
    g. Use of the EPA phosphorus procedure to exemplify the preparation of standards; X-8, 7.
    h. Preparation of the stock phosphorus solution (known composition and concentration); X-8, 8, 9, and 10.
    i. Preparation of the standard phosphorus solution (known composition and concentration); X-8, 11, 12 and 13.
    j. Preparation of a series of phosphorus solutions of known composition and concentration; X-8, 14, 15 and 16.
2. Classroom - Discuss preparation of a calibration graph using X-9, Calibration Graphs.
    a. Information needed to prepare a calibration graph; X-9, 1.
    b. Drawing and labeling the axes of a calibration graph; X-9, 2.

       (A = absorbance, C = concentration)

    c. Locating one point using absorbance concentration values. (mg P/liter is an example label for the abscissa), X-9, 3.

    d. Locating five points; X-9, 4.

    e. Connecting the five points by the line of best fit; X-9, 5.

    f. Determining the concentration of the sample using the calibration graph; X-9, 6.

3. Classroom - If the background of the participants warrants it, go through an example of calculating amounts of standard solution to be diluted to achieve calibration standards within a range given in a Method write-up.

4. Classroom - Discuss sample dilution using section V of the Training Manual outline: Use of a Spectrophotometer.

5. Classroom - Discuss the use of a spectrophotometer using XT-51: Use of the Spectronic 20 Spectrophotometer. The slide-tape is a presentation of:

    a. Warm-up

    b. Adjustments prior to use

    c. Making measurements

    d. Changing photo tube

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual Materials

X. IPW REAGENT REQUIREMENTS:

A. None

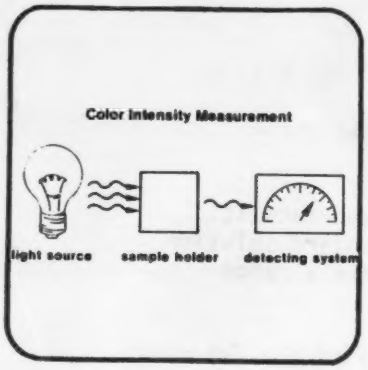
XI. DESCRIPTION OF VISUAL MATERIALS:

A. Sixteen slides, X-8: Use of a Spectrophotometer - See next pages.

B. Six slides, X-9: Calibration Graphs (Follows X-8)

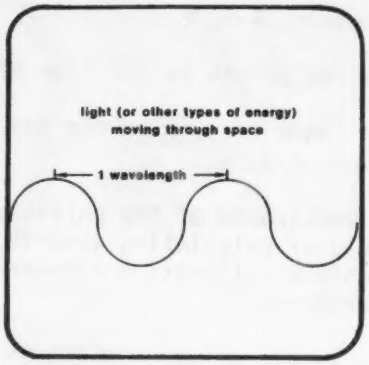
C. Thirty-five slides (also 8 minute tape), XT-51: Use of the Spectronic 20 Spectrophotometer (Follows X-9)

SERIES X-8: USE OF A SPECTROPHOTOMETER


Color Intensity Measurement

light source
sample holder
detecting system

SLIDE 1


light (or other types of energy) moving through space

1 wavelength

SLIDE 2

WAVELENGTH

- A VERY SMALL DISTANCE
- NANOMETER (nm)
- MILLIMICRON (mμ)
- COLOR INTENSITIES ARE MEASURED AT A SPECIFIC WAVELENGTH

SLIDE 3

1 nm = 0.000000001 METER

1 METER = 39.37 INCHES

SLIDE 4

LAMBERT-BEER LAW

$A = \Sigma bc$

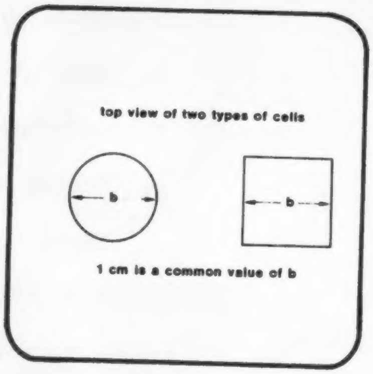
A = ABSORBANCE

Σ = A CONSTANT

b = CELL LIGHT PATH LENGTH

c = CONCENTRATION (USUALLY mg/l)

SLIDE 5


top view of two types of cells

1 cm is a common value of b

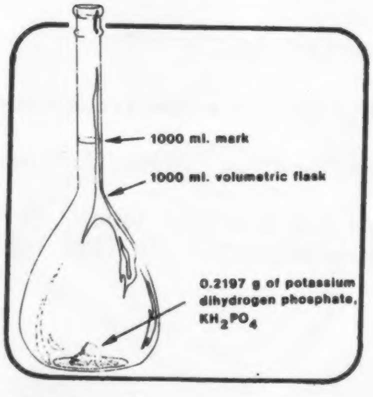
SLIDE 6

PHOSPHORUS DETERMINATION

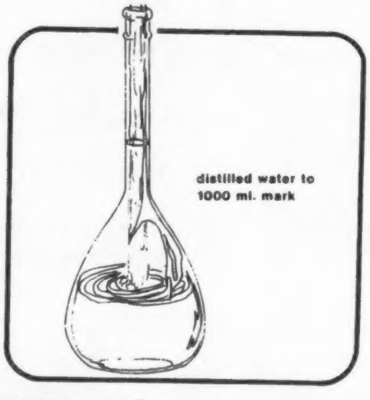
EPA METHOD

BLUE COLOR INDICATES PRESENCE OF PHOSPHORUS

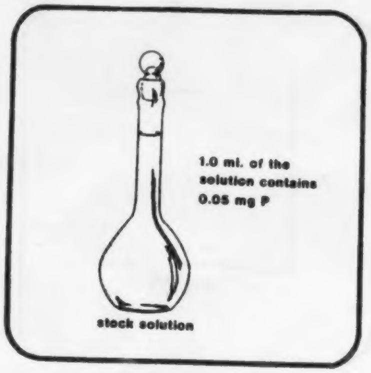
SLIDE 7


1000 ml. mark
1000 ml. volumetric flask
0.2197 g of potassium dihydrogen phosphate, KH_2PO_4

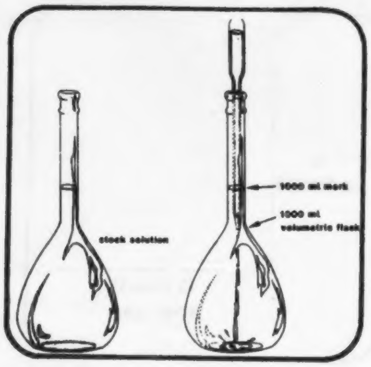
SLIDE 8


distilled water to 1000 ml. mark

SLIDE 9


1.0 ml. of the solution contains 0.05 mg P
stock solution

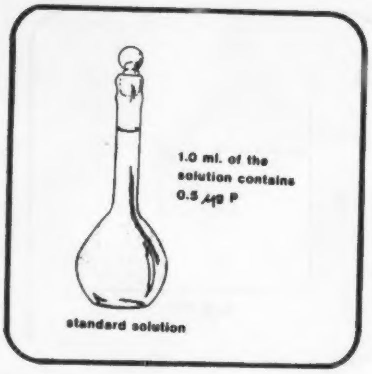
SLIDE 10


stock solution
1000 ml mark
1000 ml. volumetric flask

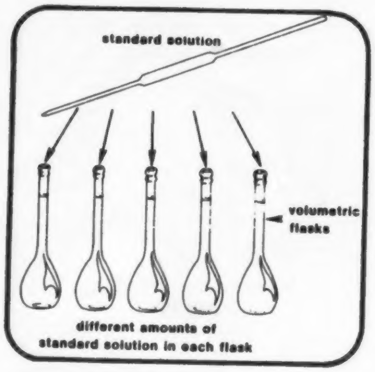
SLIDE 11

SAME AS SLIDE #9

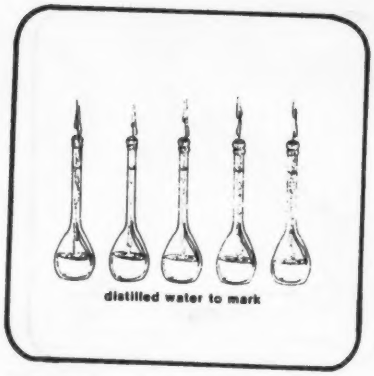
SLIDE 12


1.0 ml. of the solution contains 0.5 µg P
standard solution

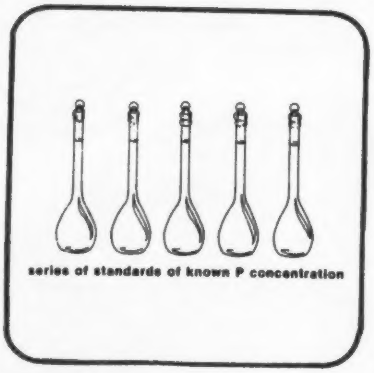
SLIDE 13


standard solution
volumetric flasks
different amounts of standard solution in each flask

SLIDE 14


distilled water to mark

SLIDE 15


series of standards of known P concentration

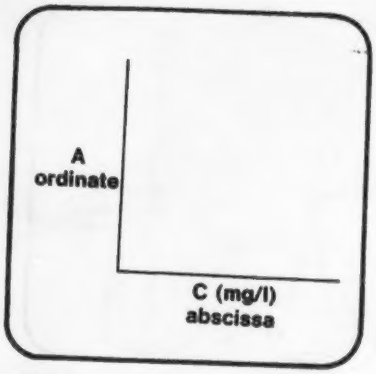
SLIDE 16

SERIES X-9: CALIBRATION GRAPHS

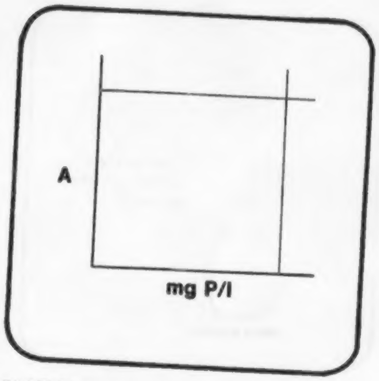
Absorbance and concentration values for each standard

Absorbance value of sample

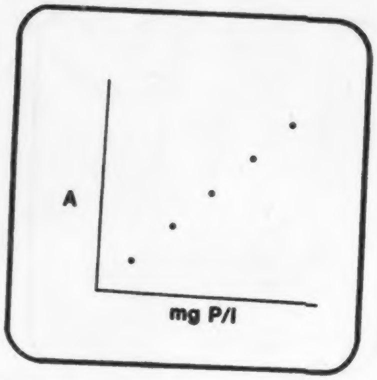
SLIDE 1



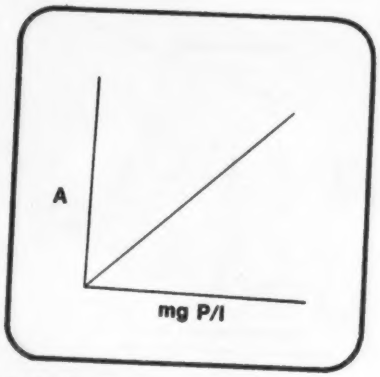
SLIDE 2



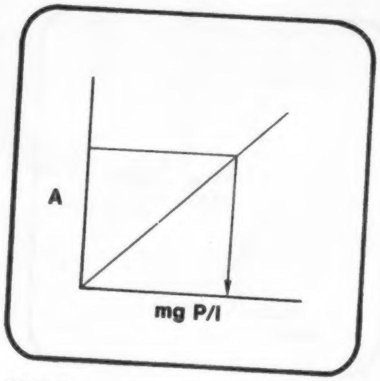
SLIDE 3



SLIDE 4



SLIDE 5



SLIDE 6

SERIES XT-51: USE OF A SPECTROPHOTOMETER

EPA Symbol

SLIDE 1

Environmental Protection
Agency
Office of Water Programs
National Training Center

SLIDE 2

Spectrophotometers

SLIDE 3

Spectronic 20

SLIDE 4

- % transmittance
- Absorbance
(optical density)

SLIDE 5

- % transmittance
- absorbance

↓

concentration

SLIDE 6

Photo
of
Spectronic 20
with
Numbers on the Controls

SLIDE 7

Photo
Featuring
# 1 - On/Off Knob

SLIDE 8

Photo
Featuring
# 2 Cell Compartment
(closed)

SLIDE 9

SERIES XT-51: USE OF A SPECTROPHOTOMETER

Photo
Featuring
# 2 - Cell Compartment
(open)

SLIDE 10

Photo
Featuring
# 3 - Read-out Scale

SLIDE 11

Photo
Featuring
# 4 - On/Off Indicator Light

SLIDE 12

Photo
Featuring
# 5 - Wavelength Scale

SLIDE 13

Photo
Featuring
# 6 - Wavelength Selection
Knob

SLIDE 14

Photo
Featuring
# 7 - Light Control
Knob

SLIDE 15

Photo
of
Rotating Wavelength
Knob

SLIDE 16

Photo
of
Turning On/Off Knob
to "On"

SLIDE 17

Photo
of
Clock to Time Warm-up

SLIDE 18

SERIES XT-51: USE OF A SPECTROPHOTOMETER

Photo
of
Adjusting to Infinite
Absorbance

SLIDE 19

Photo
of
Rinsing Sample Cell

SLIDE 20

Photo
of
Filling Sample Cell

SLIDE 21

Photo
of
Wiping Sample Cell

SLIDE 22

Photo
of
Placing Cell in
Spectrophotometer

SLIDE 23

Photo
of
Adjusting to Zero
Absorbance

SLIDE 24

Photo
of
Recording Data

SLIDE 25

Cartoon
of
Lab with Many
Samples to be Run

SLIDE 26

- Remove cell
- Rinse with distilled water
- Rinse with solution
- Fill with solution
- Dry outside of cell
- Put cell into instrument
- Record reading

SLIDE 27

SERIES XT-51: USE OF A SPECTROPHOTOMETER

340 - 625 nm

950 nm

SLIDE 28

Photo
of
Turning On/Off Knob
to "Off"

SLIDE 29

Photo
of
Opening Access Cover
Underneath Instrument

SLIDE 30

Photo
of
Removing Phototube

SLIDE 31

Photo
of
Inserting Red Filter

SLIDE 32

Spectronic 20

SLIDE 33

Credits

SLIDE 34

EPA SYMBOL

SLIDE 35

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Phosphorus

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 195 minutes (45+45+95+10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of inorganic chemistry

B. Ability to correctly use volumetric glassware

C. Ability to perform calculations involving decimals

D. Completion of the lessons (or equivalent):

1. pH

2. Use of a Spectrophotometer and Calibration Graphs

3. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent facts about phosphorus forms (see VIII B.) and will determine total phosphorus in a municipal wastewater treatment plant effluent using the procedures in the training manual (see VII A. 1. b & c). He/she will draw up a calibration curve using absorbances posted by class members and will use his/her absorbance for the sample to obtain a result. He/she will also participate in a class discussion of the topics and laboratory procedures.

B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 140 minutes laboratory time, all necessary equipment and reagents, a sample, and ten minutes for class dicussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor, and obtain a result for the sample within ± 10% of the class average (unless the reason for lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:

a. "Phosphorus in the Aqueous Environment"

b. "Laboratory Procedure for Total Phosphorus" according to 1979 EPA Methods for Chemical Analysis, p. 365.2-1

c. "Use of a Spectrophotometer"

2. Fifty-three slides, 14 minute tape and script, XT-44: The Determination of Phosphorus (See XI. Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

Caution:

a. All glassware, including that used to prepare reagents, and filtering apparatus must be cleaned using special procedures. See the training outline, "Laboratory Procedure for Total Phosphorus" section III for details.

b. Filter discs must be phosphorus-free. See section IV in the training outline for details.

2. Prepare all necessary reagents and sample as in X below.

3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.

4. Review the topic outline in the training manual and the visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Check pH meters and spectrophotometers (880 or 650 nm) for accurate operation. The spectrophotometers may need a special filter and phototube for operation at 880 or 650 nm.

7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

1. Classroom - Introduction and overview of the slide-tape presentation, XT-44: The Determination of Phosphorus.

2. Present XT-44:

a. Introduction - slides 1-5

   b. Reasons why phosphorus determinations are done - slides 6-8
   c. Phosphorus terminology, ortho-and polyphosphates - slides 9-15
   d. Overview and chemistry of the analytical method - slides 16-20
   e. Sample collection, preservation, holding time - slides 21-24
   f. Total vs. dissolved phosphorus - slide 25
   g. Digestion procedures for various forms - slides 26-33
   h. pH adjustment of digested standards and sample - slides 34-35
   i. Preparation of combined reagent and color development - slides 36-41
   j. Absorbance readings and graph - slides 42-43
   k. Care of glassware - slides 44-45
   l. Applicability of procedure - slide 46
   m. Interferences - slides 47-49
   n. Summary - credits - slides 50-53

3. Answer any questions about XT-44 topics.

4. Briefing for laboratory - class will prepare standards and sample, then begin digestion. Using the laboratory procedure in the training manual, they should do section VI. Procedures A, B, and C. Each station prepares the zero standard, one assigned standard and the sample. Stop after placing flasks on hot plates (VI. C 6). A staff member will monitor the boiling, turn off the hot plates and cover the flask mouths.

5. Laboratory-Student performance of assignment (45 minutes).

6. There can be a break of 45 minutes or up to several hours at this point. The "Use of a Spectrophotometer" could be presented during this time (See IPW on the topic).

7. Briefing for laboratory - Do section VI, D, E, F and G; i. e., pH adjustment, filtration, volume adjustment and colorimetry. Refer students to "Use of a Spectrophotometer" outline for stepwise procedure to use the instrument.

8. Laboratory-Student performance of assignment (95 minutes).

9. Classroom (10 minutes)

   a. Participants post absorbance for standards.

   b. Exclude outlier absorbance values.

   c. Sections VII and VIII of Phosphorus procedure - Each makes a "calibration curve" using posted absorbances and obtains a result using own absorbance for sample. (Stress that this method is for convenience and cannot be used for real work).

d. Discuss results and any items from the laboratory session.

e. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 2 standards (zero plus one other) and 1 sample
Filter the sample

We have the entire class do the procedure on assigned standards and the sample from the beginning through starting the digestion (manual outline VI A through C). Reagents and equipment required this far have an asterisk. Then, to minimize the number of pH meters required, we present the classroom sessions on "Use of a Spectrophotometer" and "Fluoride". The remaining laboratory work on phosphorus is done as "split" sessions, with half the class doing fluoride while half finish phosphorus.

A. For each station (no more than 2 persons per station):
CAUTION: Glassware and filtering apparatus need special cleaning (VIII A. 1. above).

1. One 100 ml beaker (for buffers)

*2. Twelve boiling beads, glass

3. One filtration apparatus, small enough to filter between 10 and 50 ml of digested solution.

4. One piece phosphorus-free filter paper (may require pre-treatment), to fit the filtration apparatus, 0.45 µm pore size (Gelman GA6 or equivalent)

*5. Three flasks, 50 ml volumetric with stoppers

*6. Three flasks, 125 ml Erlenmeyer

7. One pair forceps

*8. One funnel, to fit neck of 50 ml volumetric flask

9. One pH meter with electrode(s) in a beaker with enough 6.X buffer or distilled water to cover the tip(s). A single, combination electrode is the more convenient. Also, provide a thermometer.

*10. One 10 ml graduated pipet

*11. One 50 ml volumetric pipet (for the sample). If sample has suspended solids that may plug the pipet, use a 50 ml graduated cylinder.

*12. One pipet bulb

*13. One scoop, 0.4g capacity

*14. One squeeze bottle of phosphorus-free distilled water

*15. One marking pencil

*16. One apron for each student

*17. One pair safety glasses for each student

B. Shared:

*1. One set of volumetric pipets (1, 3, 5, 10, 20, 50 ml) per 6 stations if 1 standard is assigned to each.

*2. Enough hot plates in a hood to accommodate 3-125 ml Erlenmeyer flasks per station.

3. One spectrophotometer (for use at 880 nm or 650 nm and having a 1 cm cell) per 3 stations.

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 1979 EPA "Methods for Chemical Analysis", p. 365. 2-1, EXCEPT for A.5.*

A. Minimum Amounts per Station (no more than 2 persons per station):

1. One hundred ml pH 6.X buffer

2. One hundred ml pH 4.X buffer

3. One point two g ammonium persulfate

4. Twenty-four ml combined reagent

5. Seventy ml standard phosphorus solution (1.0 ml = 1.0 μg P) Most will use less than 70 ml. *(20 ml stock per liter for 1 cm cells)

6. Four ml 11N sulfuric acid in dropper bottle

7. Five ml 0.11N sulfuric acid in dropper bottle

8. Ten ml 10N sodium hydroxide in dropper bottle

9. Five ml 0.1N sodium hydroxide in dropper bottle

10. Fifty ml municipal wastewater treatment plant effluent for sample. If not available, use 10 ml of the stock solution per liter to make a sample containing 0.5 mg/liter phosphorus.

B. Prepare excess of each solution to allow for rinsing out glassware, waste, and re-runs.

C. A source of phosphorus-free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Fifty-three slides (with 14 minute tape), XT-44: The Determination of Phosphorus - See next pages.

SERIES XT-44: PHOSPHORUS

EPA Symbol

SLIDE 1

The Determination of Phosphorus

U.S. Environmental Protection Agency

Water Program Operations

National Training Center

SLIDE 2

Reasons
for the
Determination

SLIDE 3

Phosphorus terminology

SLIDE 4

Photo
of
EPA Chemical
Analysis Manual

SLIDE 5

Help to assess potential biological productivity

SLIDE 6

Approximately
10 micrograms/l

SLIDE 7

Drawing of "Keystone Cop" chasing man dumping phosphates into stream

SLIDE 8

PO_4^{-3}

SLIDE 9

Phosphates + heat →
Polyphosphates + water

SLIDE 10

- Cleaning Agents
- Detergents

SLIDE 11

Polyphosphates
↓ acid heat water
Orthophosphate ion

SLIDE 12

Cartoon of mad scientist dumping organic phosphorus compounds into grinder. Orthophosphates come out.

SLIDE 13

Orthophosphate Derivatives

Trisodium Phosphate

Disodium Phosphate

Monosodium Phosphate

Diammonium Phosphate

SLIDE 14

Polyphosphate Derivatives

Sodium hexametaphosphate

Sodium tripolyphosphate

Tetrasodium pyrophosphate

SLIDE 15

1. Sample collection and preservation
2. Possible filtration
3. Possible digestion
4. pH adjustment
5. Color development
6. Spectrophotometric determination

SLIDE 16

Ammonium molybdate plus potassium antimonyl tartrate plus phosphorus plus acid
↓
Antimony phosphomolybdate complex

SLIDE 17

Antimony phosphomolybdate complex plus ascorbic acid
↓
Blue complex

SLIDE 18

Cartoon of "mad" scientist staring at test tube of blue liquid.

SLIDE 19

0.01 - 0.5 mg P/l

SLIDE 20

Drawing of two hands holding sample bottles under water.

One bottle is collecting bottom deposits.

Yes No

SLIDE 21

Photo
of
Plastic and Glass
Containers

SLIDE 22

2 ml H_2SO_4

40 mg $HgCl_2/l$
Storage at 4° C

SLIDE 23

Drawing of man in laboratory looking at clock

SLIDE 24

Photo
of
Membrane Filter
Apparatus

SLIDE 25

- ammonium persulfate & sulfuric acid digestion
- sulfuric acid digestion
- no digestion

SLIDE 26

50.0 ml

SLIDE 27

Oxidizable organics and polyphosphates

↓ ammonium persulfate + sulfuric acid digestion

Orthophosphate ion

SLIDE 28

Polyphosphates and some organics

↓ sulfuric acid digestion

Orthophosphate ion

SLIDE 29

Orthophosphate

↓ No digestion

Orthophosphate

SLIDE 30

Drawing of man scratching head. In cloud above head are "polyphosphates, organic phosphates, orthophosphates"

SLIDE 31

Photo
of
Hot Plate & Clock

SLIDE 32

Photo
of
Autoclave

SLIDE 33

Adjustment of pH

- pH meter
- sodium hydroxide

SLIDE 34

50.0 ml

SLIDE 35

Photo
of
Adding Combined Reagent

SLIDE 36

- Sulfuric acid
- Potassium antimony tartrate
- Ammonium molybdate
- Ascorbic acid

SLIDE 37

- 50 ml H_2SO_4
- 5 ml $K(SbO)C_4H_4O_6 \cdot 1/2H_2O$
- Mix

SLIDE 38

- 15 ml $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$
- Mix

SLIDE 39

- 30 ml ascorbic acid
- Mix

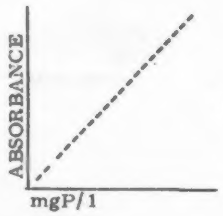
SLIDE 40

Photo
of
Flask & Clock

SLIDE 41

Photo
of
Spectrophotometer

SLIDE 42



SLIDE 43

- Hot 1:1 HCl
- Distilled water
- Color reagent
- Distilled water

SLIDE 44

Drawing of closed safe with label: P glassware

SLIDE 45

- Domestic wastes
- Industrial wastes
- Saline waters
- Surface waters
- Drinking waters

SLIDE 46

- Arsenate
- Copper
- Iron
- Silicate

SLIDE 47

Phosphorus Adsorption

- Iron
- Aluminum
- Manganese

SLIDE 48

- Salt concentration 5-20%
- Analytical error less than 1%

SLIDE 49

- Reasons for the determination
- Phosphorus terminology

SLIDE 50

Photo
of
EPA Chemical
Analysis Manual

SLIDE 51

Credit Slide

SLIDE 52

CLEAN WATER

SLIDE 53

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Determinate Error-Accuracy

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 40 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know how to control or quantitate determinate errors in laboratory results, and how to check for acceptable analytical recovery of a constituent to substantiate the validity of his/her data.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of how to use formulas involving simple calculations

B. Though not essential, it is orderly to have completed the lessons (or equivalent):

1. Sample Handling

2. Compliance Methodology

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know the difference between accuracy and precision, the causes, synonyms, effects and means to detect sources of determinate error, where to find formulas and examples of each for % recovery, % bias and % relative error, some laboratory techniques regarding accuracy, how to establish one's accuracy in performing an analysis and how to continue to monitor accuracy on a daily basis.

B. Conditions: He/she will be given the training manual and 40 minutes instruction.

C. Accepted Performance: He/she must correctly identify at least 70% of the items pertaining to the objective in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the training manual:

a. "Accuracy-Precision-Error"

b. "Elements of a Quality Assurance Program"

2. Fourteen slides, X-25: Accuracy (See XI Description of Visual Materials).

3. Five Slides, X-26: Quality Assurance (See IPW on "Elements of a Quality Assurance Program," XI Description of Visual Materials).

4. Handout: Two pages on the "Method of Standard Additions." A copy is at the end of this IPW.

5. Handout: One page on "Example of Calculating Percent Recovery for a Spike." A copy is at the end of this IPW.

6. Resource: EPA "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EMSL, Cincinnati, Ohio, 45268.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review visual materials and available handouts.

2. Prepare the lesson using the Sequencing below or your own organization of the material.

3. Duplicate copies of any handout materials.

B. Sequencing:

Slides are series X-25: Accuracy (optional: slides from X-26: Quality Assurance)

1. Introduction

   a. Before considering accuracy of results, one must have a valid sample and a skilled analyst using recognized analytical methodology.

   b. Overview: consideration of accuracy, i.e. ability to recover known quantities in chemical analyses, causes of error, how to prevent "preventable" errors and how to quantitate the effect of errors we cannot control. Statistics presented will be those most commonly used in recognized method references: percent recovery and percent bias (percent relative error).

   c. Outline, "Accuracy-Precision-Error" (A.P.E.) has details and illustrations, unless noted otherwise.

   d. Slides are from series X-25 unless otherwise noted.

2. Accuracy and Precision

   a. Analytical results are imperfect. Slide 1

   b. Define/exemplify accuracy. Results are close to the true value-standard. Accuracy can also be checked by recovery of a constituent-spike. Slide 2. Stress that both accuracy and precision are requirements for reliable data. Slide 3.

3. Error

   a. Overview: Classification + Causes of error. Determinate error has causes which can be corrected and/or quantitated. Slide 4.

   b. Synonyms for determinate error. Use A.P.E.

c. Effects of determinate error. Use A.P.E., figures 2 and 3. Slide 5

d. Detection of determinate error with unkowns - Slide 6

e. Correction of determinate error with laboratory methods. Slide 7

(1) Handout 2 pp. on "Method of Standard Additions" and discuss at point 3 on Slide 7.

4. Percent Recovery Statistic for Accuracy

a. Formula for using a standard to check percent recovery. The "observed" can be the average of several results. Slide 8

b. Formula for using a spiked sample to check percent recovery. Slide 9

c. Explanation of term, "background" in Slide 9 formula. Slide 10

d. Go through handout, "Example of Calculating Percent Recovery for a Spike" to demonstrate the correct use of the information discussed on Slides 9 and 10.

e. Checking own accuracy using a spiked sample. Slide 11. Note that one can use a standard instead. The latter calculations are easier.

f. OPTIONAL: X-26, Slide 4 shows a percent recovery control chart for monitoring accuracy on a daily basis.

5. Percent Bias (Percent Relative Error) Statistic for accuracy

a. Two major reference manuals use different names for this statistic. Slide 12

b. Formula for using a standard to check percent bias. Slide 13

c. OPTIONAL: X-26, Slide 5 shows a percent bias control chart for monitoring accuracy on a daily basis.

6. Relationship of Percent Recovery and Percent Bias Statistics. Slide 14 (last slide).

7. Daily Application

a. Suggested (EPA Handbook for AQC) - 10% of tests be spikes to check accuracy.

8. Summary

a. Again stress valid sample and skilled analyst using recognized analytical methodology are prior to checking accuracy.

b. Summarize presentation.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual materials

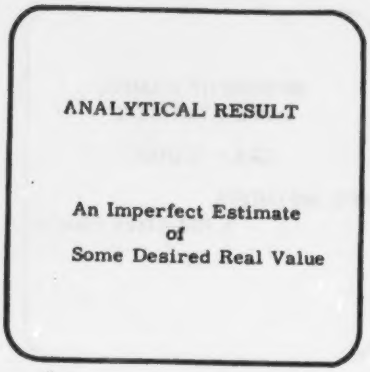
X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS

A. Fourteen slides, X-25: Accuracy - See next pages.

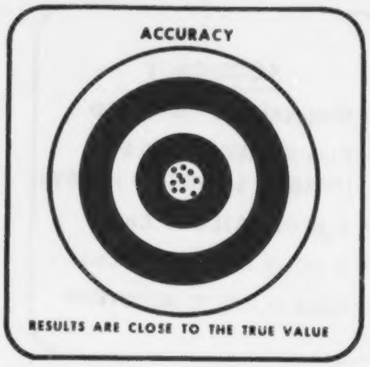
SERIES X-25: ACCURACY



ANALYTICAL RESULT

An Imperfect Estimate
of
Some Desired Real Value

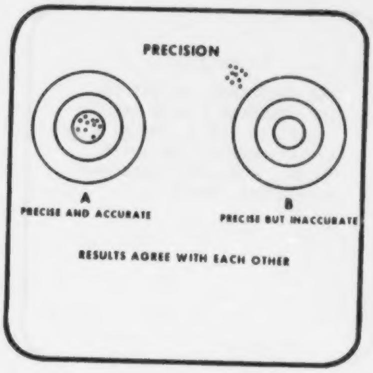
SLIDE 1



ACCURACY

RESULTS ARE CLOSE TO THE TRUE VALUE

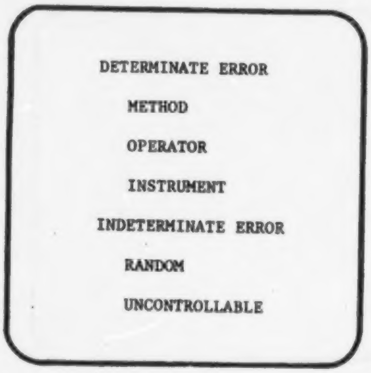
SLIDE 2



PRECISION

RESULTS AGREE WITH EACH OTHER

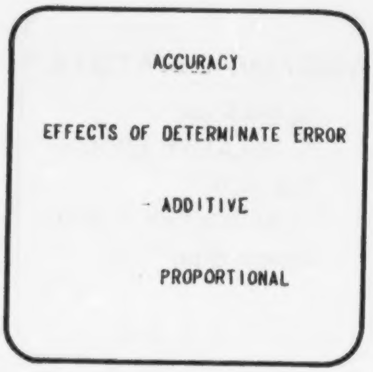
SLIDE 3



DETERMINATE ERROR
- METHOD
- OPERATOR
- INSTRUMENT

INDETERMINATE ERROR
- RANDOM
- UNCONTROLLABLE

SLIDE 4



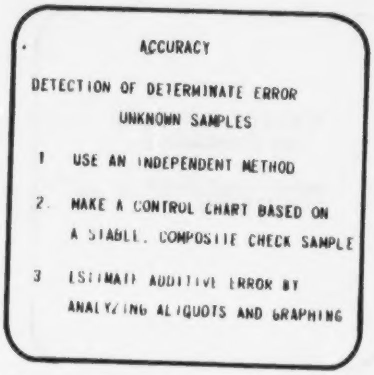
ACCURACY

EFFECTS OF DETERMINATE ERROR

- ADDITIVE

PROPORTIONAL

SLIDE 5

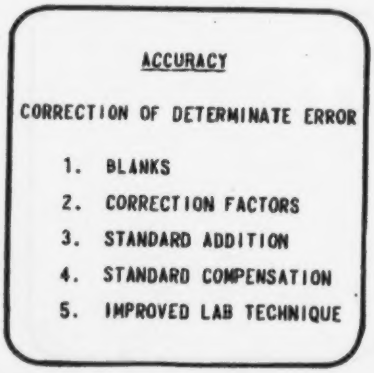


ACCURACY

DETECTION OF DETERMINATE ERROR
UNKNOWN SAMPLES

1 USE AN INDEPENDENT METHOD
2. MAKE A CONTROL CHART BASED ON A STABLE, COMPOSITE CHECK SAMPLE
3 ESTIMATE ADDITIVE ERROR BY ANALYZING ALIQUOTS AND GRAPHING

SLIDE 6

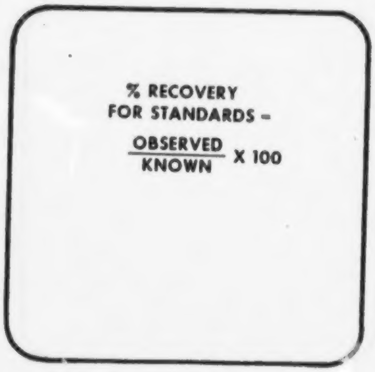


ACCURACY

CORRECTION OF DETERMINATE ERROR

1. BLANKS
2. CORRECTION FACTORS
3. STANDARD ADDITION
4. STANDARD COMPENSATION
5. IMPROVED LAB TECHNIQUE

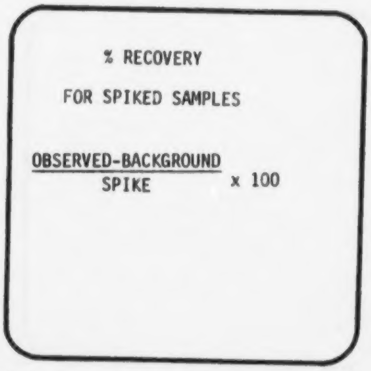
SLIDE 7



% RECOVERY
FOR STANDARDS -

$$\frac{\text{OBSERVED}}{\text{KNOWN}} \times 100$$

SLIDE 8



% RECOVERY
FOR SPIKED SAMPLES

$$\frac{\text{OBSERVED-BACKGROUND}}{\text{SPIKE}} \times 100$$

SLIDE 9

SERIES X-25: ACCURACY

"BACKGROUND"

The analytical result for the unspiked sample with regard to its volume as a constituent of the spike which yields the "observed" analytical result.

SLIDE 10

ACCURACY

- RUN SAMPLE UNSPIKED
- RUN SPIKED SAMPLE (DOUBLE LOWEST, I to 75%)
- 7 REPLICATES of EACH
- 2 HOUR LAB TIME (min.)
- CALCULATE % RECOVERY

SLIDE 11

DIFFERENT NAMES,
SAME STATISTIC:

EPA - % BIAS

ST'D METHODS
- % RELATIVE ERROR

SLIDE 12

% BIAS (% relative error)
FOR STANDARDS =

$$\frac{\text{MEAN - TRUE VALUE}}{\text{TRUE VALUE}} \times 100$$

SLIDE 13

ACCURACY STATISTICS

% BIAS OR
% RELATIVE ERROR
EQUALS
% RECOVERY – 100
(gives sign)

SLIDE 14 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

8.5* Method of Standard Additions: In this method, equal volumes of sample are added to a deionized distilled water blank and to three standards containing different known amounts of the test element. The final volumes of all the solutions must be the same. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations from the weight of analyte added to each of the original volumes of sample plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1.

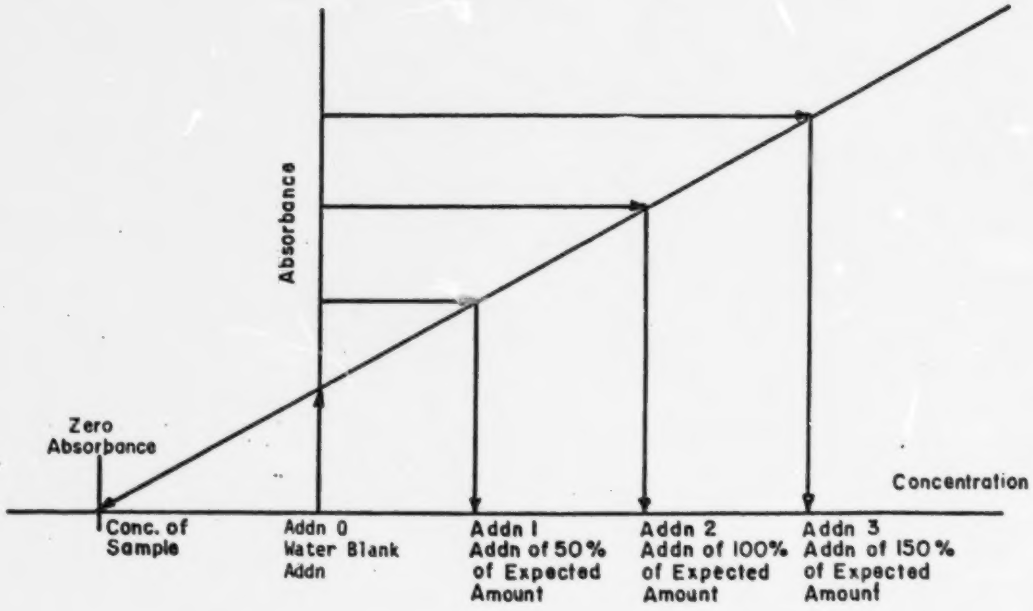


FIGURE 1. STANDARD ADDITION PLOT

The method of standard additions can be very useful, however, for the results to be valid the following limitations must be taken into consideration:

a) the absorbance plot of sample and standards must be linear over the concentration range of concern. For best results the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%) caution should be exercised.

---

*from: 1979 EPA "Methods for Chemical Analysis of Water and Wastes"

b) the effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes and the standard addition should respond in a similar manner as the analyte.

c) the determination must be free of spectral interference and corrected for non-specific background interference.

EXAMPLE OF CALCULATING PERCENT RECOVERY FOR A SPIKE

A sample was collected and a 25 ml aliquot was analyzed for nitrate nitrogen. The result of the analysis was 500 µg/liter. A 25 ml spiked aliquot of the same sample was prepared using 1 ml of a spike solution which contained 10 mg/liter nitrate nitrogen. The result of analyzing the spiked sample was 898 µg/liter. Calculate the percent recovery.

$$P = \frac{\text{observed} - \text{background}}{\text{spike amount}} \times 100$$

$$\text{observed is } \left(\frac{898\ \mu g}{\text{liter}}\right) (0.025 \text{ liter}) = 22.45\ \mu g$$

$$\text{background is } \left(\frac{500\ \mu g}{\text{liter}}\right) (0.024 \text{ liter}) = 12.00\ \mu g$$

$$\text{spike amount is } \left(\frac{10{,}000\ \mu g}{\text{liter}}\right) (0.001 \text{ liter}) = 10.00\ \mu g$$

$$P = \frac{22.45\ \mu g - 12.00\ \mu g}{10.00\ \mu g} \times 100$$

$$P = 104.5\%$$

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Fluoride

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 160 minutes (60+95+5)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of inorganic chemistry

B. Ability to use volumetric glassware

C. Completion of the lessons (or equivalent):

1. Use of a Spectrophotometer and Calibration Graphs

2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent facts about fluoride, the manual distillation procedure and four analytical procedures for determining fluoride (see VIII B.). He/she will test four samples by the SPADNS (by calculation) and the electrode methods (8tests) using the procedures in the training manual (see VII. A. 1. b and c) and will report the results. He/she will also participate in a class discussion of the laboratory procedures and the topic in general.

B. Conditions: He/she will be given the training manual, 60 minutes of instruction, 95 minutes laboratory time, all necessary equipment and reagents, four samples and five minutes for final questions.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor and obtain results within ± 0.1 mg/liter of the results obtained by the Instructor (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:

a. "Control of Interfering Ions in Fluoride Determinations"

   b. "Fluoride Analytical Procedures - SPADNS" according to 14th ed. Standard Methods, p. 393.

   c. "Fluoride Analytical Procedures - Electrode" according to Orion Research, Inc. instrument directions. For other instruments, use the manufacturer's operating instructions. Also see 14th ed. Standard Methods, p. 391.

2. Forty-six slides and 16 minute tape, XT-80: Fluoride Analytical Procedures (see XI., Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.
2. Prepare all necessary reagents and samples as in X below.
3. Review the topic outline in the manual, then the laboratory procedures by performing the student assignment using the outlines in the training manual. Notice that two of the samples contain interferences for the SPADNS method to exemplify interfering ions.
4. If necessary, review other aspects of the topic by consulting Standard Methods, 14th ed, Section 414, Fluoride. Also review the visual materials (see XI below).
5. Prepare the lesson using the Sequencing below or your own organization of the material.
6. Check specific ion meters (and/or pH meters), the electrodes and the spectrophotometers (570 nm) for accurate operation.
7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

1. Classroom - Introduction
   a. Existence of fluoride in waters
   b. How fluoride ions are introduced
2. Classroom - Introduce and present XT-80: Fluoride Analytical Procedures. Tell the participants about the information to expect during the presentation:
   a. (Title and Credits, slides 1-4)
   b. Fluoride and dental effects (slides 5-6)

c. Titles and overview of four methods approved for analysis of drinking water samples (slides 7-14)

d. Stepwise procedure and calculations for the SPADNS method, (slides 15-21)

e. Stepwise procedure using the electrode method (slides 22-30)

f. Interferences in fluoride analyses (slides 31-32)

g. Steps and apparatus for the distillation procedure to remove interferences before analysis (slides 33-39)

h. Summary of information presented (slides 40-45)

i. (Logo, slide 46)

3. Classroom - Discuss approved methods. Differentiate between water supply regulations and NPDES regulations. Section IV in the training manual outline, "Control of Interfering Ions, etc," is a summary of Compliance Methodology.

   a. Electrode (Distillation may be required for NPDES).

   b. Distillation and SPADNS

   c. Automated complexone (Method includes distillation).

   d. Distillation and Eriochrome cyanine R (only approved for drinking water samples)

4. Classroom - Summary

5. Laboratory - 95 minutes

   a. Briefing and assign four samples to be tested by the SPADNS and also by the electrode method (8 tests in all). If every other station begins with the SPADNS test and every other begins with the electrode method, the required spectrophotometers and electrode-meter apparatus can be shared, one per two stations.

   b. Student performance of assignment.

   c. Students report results.

6. Classroom Final Discussion - 5 minutes

   a. Any items from laboratory session

   b. Resolve any questions

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Four samples to analyze by two methods - SPADNS and an electrode with selective ion meter. (If a pH meter is to be used, a blank and three standards for calibration should be assigned). To minimize the number of spectrophotometers and electrodes with meters required, the laboratory session could be conducted twice with half the class doing another procedure requiring about the same time, e.g., Phosphorus pH adjustment and colorimetry.

A. For each station (no more than two persons per station):

1. Six 150 ml plastic beakers (eight for pH meter)
2. Six 125 ml Erlenmeyer flasks
3. Three 10 ml volumetric pipets
4. One 50 ml volumetric pipet
5. One pipet bulb
6. One squeeze bottle fluoride-free distilled water
7. One marking pencil
8. One apron for each student
9. One pair safety glasses for each student
10. Optional: Magnetic stirring apparatus

B. Shared:

1. One spectrophotometer for use at 570 nm and having a 1 cm cell per two stations (see VIII. B. 5. a.).
2. One fluoride specific ion electrode and meter per two stations (see VIII. B. 5. a.).
3. One stop watch or timing device.

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition "Standard Methods", page 392 and page 393.

A. Minimum amounts per station (no more than two persons per station):

1. Fifty ml distilled water
2. Three hundred ml TISAB (400 ml for pH meter)
3. One hundred ml fluoride standard, 1 mg/liter F^- (selective ion meter and SPADNS)
4. One hundred ml fluoride standard, 2 mg/liter F^- (only if a pH meter is used)
5. Sixty ml fluoride standard, 10 mg/liter F^- (selective ion meter)
6. Seventy-five ml SPADNS reagent

7. One hundred ml sample #1 containing 0.55 mg/liter F^- (5.5 ml fluoride stock solution per liter) Both of the methods (SPADNS and electrode) give correct results for this sample.

8. One hundred ml sample #2 containing 1.05 mg/liter F^- (10.5 ml fluoride stock solution per liter). Both of the methods give correct results for this sample.

9. One hundred ml sample #3 containing 0.75 mg/liter F^- plus 300 mg/L sulfate (7.5 ml fluoride stock solution plus 0.4437g anhydrous sodium sulfate per liter). Only the electrode gives correct results for this sample.

10. One hundred ml sample #4 containing 0.95 mg/liter F^- plus 3 mg/L aluminum (9.5 ml fluoride stock solution plus 52.752 mg aluminum potassium sulfate with $\cdot 12H_2O$). Only the electrode gives correct results for this sample.

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of fluoride-free distilled water should be available during laboratory sessions.

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Forty-six slides (with 17 minute tape), XT-80: Fluoride Analytical Procedures. See next pages.

EPA

PRESENTS

SLIDE 1

FLUORIDE ANALYTICAL PROCEDURES

SLIDE 2

PREPARED BY:

EPA
OWP
MOTD
NTOTC

SLIDE 3

TECHNICAL DIRECTION BY:

JOHN PFAFF
ERVIN BELLACK
THOMAS HUSHOWER

SLIDE 4

PHOTO

CHILD DRINKING WATER FROM FOUNTAIN

SLIDE 5

PHOTO

OPERATOR IN LAB CONDUCTING ANALYSIS

SLIDE 6

LIST OF EPA - APPROVED METHODS AND THE RELATED REFERENCES

SLIDE 7

AUTOMATED COMPLEXONE METHOD

NOTABLE FEATURES

SLIDE 8

ZIRCONIUM-ERIOCHROME CYANINE R METHOD

NOTABLE FEATURES

SLIDE 9

SPADNS WITH DISTILLATION
AND THE
ELECTRODE METHODS

SLIDE 10

THE SPADNS ZIRCONIUM FLUORIDE
REACTION

SLIDE 11

PHOTO
ANALYST USING A SPEC 20

SLIDE 12

DRAWING OF ELECTRODE
IMMERSED IN WATER

SLIDE 13

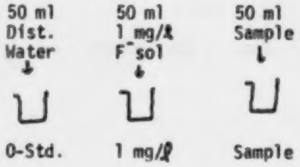
PHOTO
ANALYST USING ION METER

SLIDE 14

TITLE
SPADNS PHOTOMETRIC METHOD

SLIDE 15

Preparing Standards & Sample
SPADNS Procedure


50 ml
Dist.
Water
50 ml
1 mg/ℓ
F⁻ sol
50 ml
Sample
0-Std.
1 mg/ℓ
Sample

SLIDE 16

PHOTO
ANALYST ADDING REAGENT

SLIDE 17

PHOTO
CLOSE-UP OF SPEC 20 SHOWING
WAVELENGTH ADJUSTMENT

SLIDE 18

PHOTO

ANALYST USING SPECTRONIC 20

SLIDE 19

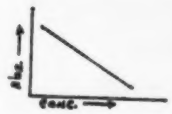
ABSORBANCE EQUATION

$$X = \frac{A_0 - A_x}{A_0 - A_1} = mg/\ell$$

TERMS DEFINED

SLIDE 20

Graph of Absorbance vs.Conc.



SLIDE 21

TITLE

THE ELECTRODE METHOD

SLIDE 22

PHOTO

ANALYST USING AN EXPANDED SCALE pH METER

SLIDE 23

PHOTO

ANALYST USING AN ION METER

SLIDE 24

Preparing Standards & Samples
Electrode Procedure

10 ml of 1.0 mg/ℓ	10 ml of 10.0 mg/ℓ	10 ml of sample

3- 100 ml plastic beakers

SLIDE 25

PHOTO

ANALYST ADDING TISAB

SLIDE 26

PHOTO

ANALYST CALIBRATING METER

SLIDE 27

SERIES XT-80: FLUORIDE ANALYTICAL PROCEDURES

PHOTO
ANALYST ADJUSTING TEMPERATURE
COMPENSATOR KNOB

SLIDE 28

PHOTO
ANALYST READING SAMPLE

SLIDE 29

PHOTO
SET-UP WITH MAGNETIC STIRRER

SLIDE 30

LIST: INTERFERING SUBSTANCES IN
FLUORIDE ANALYSIS

SLIDE 31

SPADNS METHOD INTERFERENCES
LIST: INTERFERING SUBSTANCES

SLIDE 32

TITLE
REMOVAL OF INTERFERENCES BY
DISTILLATION

SLIDE 33

DISTILLATION REACTION EQUATION

SLIDE 34

PHOTO
ANALYST WITH
DISTILLATION SET-UP

SLIDE 35

PHOTO
DISTILLATION IN PROGRESS

SLIDE 36

PHOTO

ANALYST ADDING SAMPLE TO FLASK

SLIDE 37

PHOTO

CLOSE-UP OF THE DISTILLATE RECEIVER

SLIDE 38

MOST PREVALENT INTERFERING SUBSTANCES:

ALUMINUM
HEXAMETAPHOSPHATE

SLIDE 39

PHOTO

SAME AS SLIDE #5

CHILD DRINKING WATER FROM A FOUNTAIN

SLIDE 40

SAME AS SLIDE #7

LIST OF EPA-APPROVED METHODS AND THE RELATED REFERENCES

SLIDE 41

PHOTO

SAME AS SLIDE #12

ANALYST USING A SPEC 20

SLIDE 42

PHOTO

DISTILLATION IN PROGRESS

SAME AS SLIDE #36

SLIDE 43

PHOTO

ANALYST USING ION METER

SAME AS SLIDE #24

SLIDE 44

PHOTO

OPERATOR IN LAB DOING ANALYSIS

SLIDE 45

SAFE DRINKING

WATER EMBLEM

SLIDE 46

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Nitrate and Nitrite Nitrogen

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 310 minutes (30+10+90+30+105+45)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of inorganic chemistry

B. Ability to correctly use volumetric glassware

C. Ability to perform calculations involving decimals

D. Completion of the lessons (or equivalent):

1. pH

2. Use of a Spectrophotometer and Calibration Graphs

3. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent facts about nitrate and nitrite (see VIII B). Using the procedures in the course manual (see VII A 1 b and c), he/she will prepare a cadmium reduction column, process a blank and assigned standard or sample through it and obtain absorbance readings. He/she will also process a non-reduced blank and assigned standard or sample and obtain absorbance readings. Using his/her own absorbance values, the participant will calculate the efficiency of his/her column. He/she will use results pooled from the class and construct calibration curves (2) for reduced and non-reduced standards, then use them to obtain results for the samples. He/she will calculate NO_3-N from these results and will also learn how to express results as NO_3 or as NO_2. He/she will give the completed graphs to the Instructor. He/she will also participate in a class discussion of the topics and laboratory procedures.

B. Conditions: He/she will be given the training manual, 70 minutes of instruction, 195 minutes laboratory time, all necessary equipment and reagents, a sample, and 45 minutes for drawing up graphs, calculations, and class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor, and obtain results for the sample within ± 15% of the theoretical answers provided by the Instructor (unless the reason for a lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:
   a. "Ammonia, Nitrites and Nitrates"
   b. "Determination of Nitrate/Nitrite Nitrogen (Cadmium Reduction Method)" according to 1974 EPA Methods for Chemical Analysis, page 201.
   C. "Use of a Spectrophotometer"
2. Twenty slides, X-23: Nitrate/Nitrite Nitrogen (See XI Description of Visual Materials)

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.
2. Prepare all necessary reagents and sample as in X below.
3. Review the laboratory procedure by performing the student assignment using the outline in the training manual. It is suggested that the Instructor prepare a few columns for back-up if participants let theirs go dry, etc., when it is too late to start over.
4. Review the topic outline in the training manual and the visual materials. See XI below.
5. Prepare the lesson using the Sequencing below or your own organization of the material.
6. Check pH meters and spectrophotometers (540nm) for accurate operation.
7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

Slides are series X-23: Nitrate/Nitrite Nitrogen

1. Classroom - Analysis of Nitrate and Nitrite Nitrogen (30 minutes) Use outline on "Ammonia, Nitrites and Nitrates"
   a. Introduction
      1) Nitrogen Cycle, note conversions, sources of nitrate and nitrite - Slide 1.

2) Types of nitrogen analyses, desired forms - Slide 2.

b. Nitrite

1) Sources (from nitrogen cycle), bacterial oxidation of ammonia or reduction of nitrate

2) Analytical Method - Slide 3.

3) Scope, Range, Preservation - Slide 4.

4) Procedure Steps - Slide 5.

5) Interferences - Slide 6.

6) Compliance Methodology in training manual. Diazotization is the only method cited as of 8/80.

c. Nitrate

1) Source (from nitrogen cycle), bacterial oxidation of nitrite, two mechanisms from atmospheric nitrogen

2) Analytical Methods - Slide 7.

3) Sample Preservation in training manual, Section II

4) Brucine Sulfate Method, procedure and interferences - Slide 8 and 9.

5) Automated Hydrazine Reduction Method - Slide 10.

6) Note: Class will do the third method, cadmium reduction, in the laboratory. It is presented next as a main topic. Give an overview of it now since it utilizes the same approach as hydrazine reduction - Slide 11

7) Compliance Methodology in training manual, Section V. D., as of 8/80.

2. Classroom - Cadmium Reduction Columns (10 minutes) Use outline on "Determination of Nitrate/Nitrite Nitrogen".

a. Students are to prepare the cadmium and pack a column - Slide 12.

b. Steps involved in preparation, stress the steps - Slide 13.

c. Briefing on laboratory procedure in training manual, Sections II, III, IV.

1) Note Section II on apparatus has details on making a column.

2) Section III has reagent preparations. The reagents are made up.

3) Note Section IV, division titles for overview. Note on copper sulfate treatment that one addition is usually enough to get the brown copper precipitate. Make any other comments on the laboratory work.

        4) Remarks on toxicity of cadmium

3. Laboratory - Student performance of assignment, all of Section IV (90 minutes).

4. Note: There can be a break of unlimited time at this point.

5. Classroom (5 minutes)

    a. Follow up on preparation-of-column laboratory session, discussing any occurrences in the laboratory of general instructional interest.

    b. Interferences to column function. Note Section V in manual - Slide 14.

6. Classroom - Determination of NO_2 + NO_3-N and of NO_2-N using the column. (25 minutes)

    a. Role of ammonium chloride to provide the H^+ - Slide 15.

    b. Procedure steps for NO_2 + NO_3-N, - Slide 16.

    c. Procedure steps for NO_2-N are the same asfor NO_2 + NO_3-N, except for the reduction step - Slide 17.

    d. Briefing on procedures in manual, Sections VI and VII. Have participants circle the roman numeral and capital letter division titles to impress on them the divisions of the procedures.

        1) Assign blank, and either a nitrate standard or the sample for each station.

        2) Assign blank, and either a nitrite standard or the sample for each station. For standards, assign the same concentration as assigned for nitrate.

        3) Refer students to "Use of a Spectrophotometer" outline for stepwise procedures to use this instrument for VI.D. and VII.D.

        4) They cannot do calibration curves (VI.E. and VII.E) until results are posted.

7. Laboratory - Student performance of assignment (105 minutes)

8. Class Calculations, Graphing, Discussion (45 minutes)

    a. Participants post absorbance value for nitrate and for nitrite standards.

    b. Discuss and have participants calculate their column efficiency according to formula in manual procedure, Section VIII - Slide 18. (Those who analyzed the sample should use the results obtained by someone who did a pair of standards).

c. Discuss posted results. Discard questionable values.

d. Copies of arithmetic graph paper are in the training manual.

e. Assignment:

    1) Participants construct a graph for the reduced nitrate standards and a graph for nitrite standards - Slide 19 and 20. (Some may need assistance).

    2) They use their absorbance values for their reduced sample on one graph and for their non-reduced sample on the other to obtain concentrations for each.

    3) They calculate NO_3-N in the sample by subtraction.

f. Participants report results. Announce theoretical "true" results (if known).

g. Use manual, Section IX in procedure to show how to calculate NO_3 or NO_2 from the data. Have participants do this, using theoretical results.

h. Summary Remarks on the Procedure

    1) column rate, checking efficiency, re-activation, advantage of duplicates

    2) cautions on cadmium disposal

    3) preservation and holding times for samples (Ammonia, etc. outline, Section II).

i. Collect graphs. These can be returned later after Instructor checks them.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Prepare and activate a reduction column.
1 blank and either one nitrate standard or the sample
1 blank and either one nitrite standard of the same concentration as assigned for nitrate, or the sample

A. For each station (no more than two persons per station):

1. Two beakers, 150 ml
2. Two beakers, 250 ml
3. One beaker, 400 ml
4. One cut-off 100 ml pipet (for reduction) mounted on a stand
5. One cylinder, graduated, 50 ml
6. One cylinder, graduated, 100 ml
7. Four flasks, 250 ml Erlenmeyer
8. Three flasks, 100 ml volumetric with stoppers

9. One very large funnel containing fluted filter paper (for Cd rinsings) set in an old 2-3 liter bottle
10. Small wad glass wool for column
11. One 3 inch length rubber hose (4 cm ID)
12. One paper clip
13. One 25 ml volumetric pipet
14. One to three 50 ml volumetric pipets
15. One propipet bulb
16. One screw-type clamp to regulate column flow
17. One snap-type clamp to start and stop flow
18. One 60 mesh sieve
19. One spatula or stirring rod to transfer moist cadmium
20. One squeeze bottle of nitrate-nitrite-free distilled water
21. One marking pencil
22. One apron for each student
23. One pair of safety glasses for each student

B. Shared:

1. One pH meter per 2 stations. Each should have the electrode(s) in a beaker with enough 6.X buffer or distilled water to cover the tip(s). Also, one thermometer for each meter.
2. Two sets of volumetric pipets (1, 2, 5, 10 ml) per 4 stations
3. Two 1 or 2 ml pipets graduated at 0.5 ml per 4 stations
4. One 10 ml graduated pipet for each "common" beaker of color reagent
5. One spectrophotometer (for use at 540 nm and having a 1 cm cell) per 3 stations

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 1974 EPA "Methods for Chemical Analysis," page 202.

A. Minimum amounts per station (no more than two persons per station):

1. 610 ml concentrated ammonium chloride - EDTA solution. Use 210 ml of this to prepare 350 ml dilute ammonium chloride - EDTA solution to prepare the column. The remaining 400 ml is used for the test procedure.
2. Eight ml color reagent
3. ∿ 1 ml concentrated hydrochloric acid in dropper bottle
4. ∿ 1 ml concentrated ammonium hydroxide in a dropper bottle
5. Sixty ml 6 N hydrochloric acid

6. Two hundred ml 2% copper sulfate solution

7. ∿ 20 g cadmium, 40-60 mesh. Pre-weigh into container for stations.

8. ∿ 100 ml pH 4.X buffer

9. ∿ 100 ml pH 6.X buffer

10. Twenty-five ml 1.00 mg NO_3 - N/liter (to activate column)
    UNSTABLE - Prepare just before use by diluting 1 ml stock potassium nitrate solution to one liter.

11. Fifteen ml potassium nitrate standard solution
    UNSTABLE - Prepare just before use by diluting 10 ml stock potassium nitrate solution to one liter.

12. Fifteen ml potassium nitrite standard solution
    UNSTABLE - Prepare just before use by diluting 10 ml stock potassium nitrite solution to one liter.

13. Fifty ml sample containing up to 1 mg NO_2 + NO_3 - N/liter
    Example: 40 ml of nitrate standard plus 40 ml nitrite standard solutions per liter gives 0.4 mg /liter of each form for a total of 0.8 mg NO_2 + NO_3 - N/liter. A municipal wastewater treatment plant effluent known to contain preferably both forms can be used for a sample. You may need to assign dilutions so test is applicable.

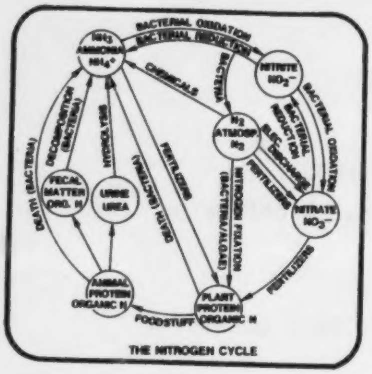
B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of nitrate-nitrite-free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

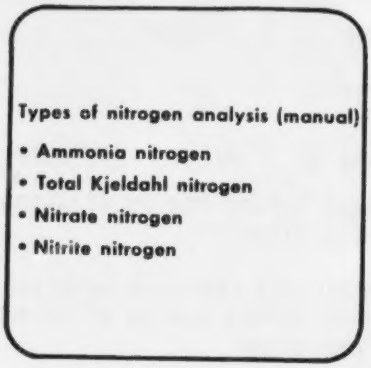
A. Twenty slides, X-23 Nitrate/Nitrite Nitrogen - See next pages.

SERIES X-23: NITRATE/NITRITE NITROGEN


NH3
AMMONIA
NH4+
BACTERIAL OXIDATION
BACTERIAL REDUCTION
NITRITE
NO2−
CHEMICALS
BACTERIA
N2
ATMOSP
N2
BACTERIAL REDUCTION
BACTERIAL OXIDATION
ELEC. DISCHARGE
FERTILIZERS
NITRATE
NO3−
DEATH (BACTERIA)
DECOMPOSITION (BACTERIA)
HYDROLYSIS
FERTILIZERS
DEATH (BACTERIA)
NITROGEN FIXATION (BACTERIA/ALGAE)
FECAL MATTER ORG. N
URINE UREA
FERTILIZERS
ANIMAL PROTEIN ORGANIC N
PLANT PROTEIN ORGANIC N
FOODSTUFF

THE NITROGEN CYCLE

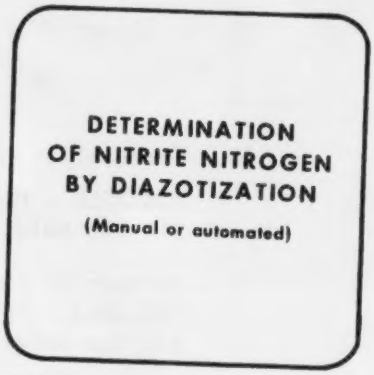
SLIDE 1



Types of nitrogen analysis (manual)

- Ammonia nitrogen
- Total Kjeldahl nitrogen
- Nitrate nitrogen
- Nitrite nitrogen

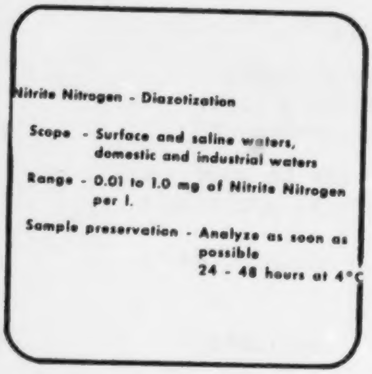
SLIDE 2



DETERMINATION
OF NITRITE NITROGEN
BY DIAZOTIZATION

(Manual or automated)

SLIDE 3



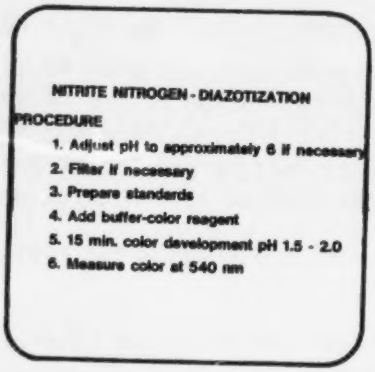
Nitrite Nitrogen - Diazotization

Scope - Surface and saline waters, domestic and industrial waters

Range - 0.01 to 1.0 mg of Nitrite Nitrogen per l.

Sample preservation - Analyze as soon as possible
24 - 48 hours at 4°C

SLIDE 4

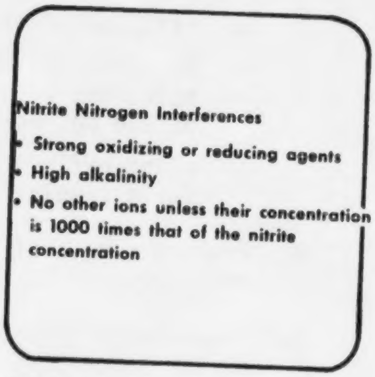


NITRITE NITROGEN - DIAZOTIZATION

PROCEDURE

1. Adjust pH to approximately 6 if necessary
2. Filter if necessary
3. Prepare standards
4. Add buffer-color reagent
5. 15 min. color development pH 1.5 - 2.0
6. Measure color at 540 nm

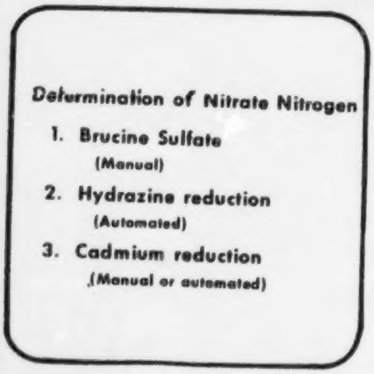
SLIDE 5



Nitrite Nitrogen Interferences

- Strong oxidizing or reducing agents
- High alkalinity
- No other ions unless their concentration is 1000 times that of the nitrite concentration

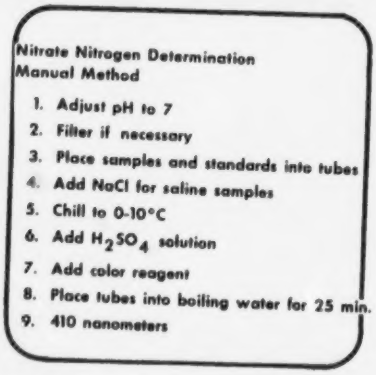
SLIDE 6



Determination of Nitrate Nitrogen

1. Brucine Sulfate
   (Manual)
2. Hydrazine reduction
   (Automated)
3. Cadmium reduction
   (Manual or automated)

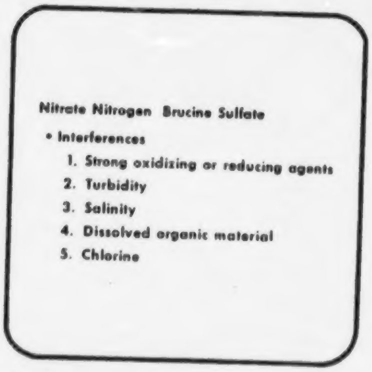
SLIDE 7



Nitrate Nitrogen Determination
Manual Method

1. Adjust pH to 7
2. Filter if necessary
3. Place samples and standards into tubes
4. Add NaCl for saline samples
5. Chill to 0-10°C
6. Add H_2SO_4 solution
7. Add color reagent
8. Place tubes into boiling water for 25 min.
9. 410 nanometers

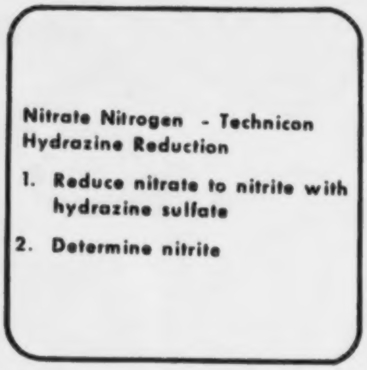
SLIDE 8



Nitrate Nitrogen Brucine Sulfate

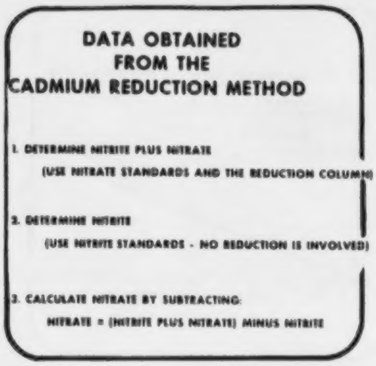
- Interferences
  1. Strong oxidizing or reducing agents
  2. Turbidity
  3. Salinity
  4. Dissolved organic material
  5. Chlorine

SLIDE 9


Nitrate Nitrogen - Technicon Hydrazine Reduction

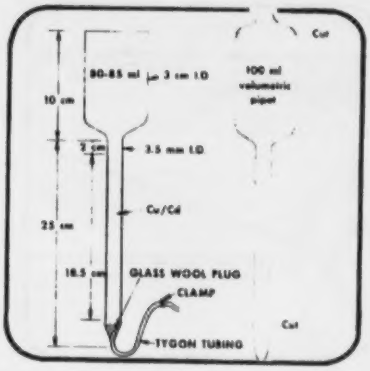
1. Reduce nitrate to nitrite with hydrazine sulfate
2. Determine nitrite

SLIDE 10

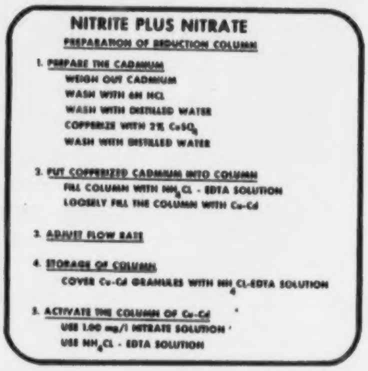

DATA OBTAINED
FROM THE
CADMIUM REDUCTION METHOD

1. DETERMINE NITRITE PLUS NITRATE
   (USE NITRATE STANDARDS AND THE REDUCTION COLUMN)
2. DETERMINE NITRITE
   (USE NITRITE STANDARDS - NO REDUCTION IS INVOLVED)
3. CALCULATE NITRATE BY SUBTRACTING:
   NITRATE = (NITRITE PLUS NITRATE) MINUS NITRITE

SLIDE 11

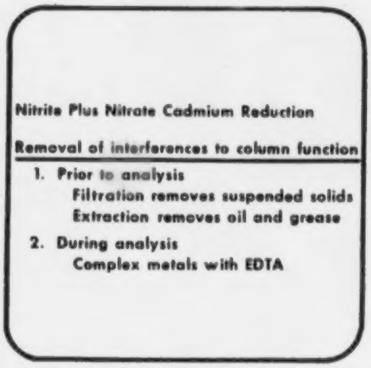


SLIDE 12


NITRITE PLUS NITRATE
PREPARATION OF REDUCTION COLUMN

1. PREPARE THE CADMIUM
   WEIGH OUT CADMIUM
   WASH WITH 6N HCL
   WASH WITH DISTILLED WATER
   COPPERIZE WITH 2% $CuSO_4$
   WASH WITH DISTILLED WATER
2. PUT COPPERIZED CADMIUM INTO COLUMN
   FILL COLUMN WITH NH_4CL - EDTA SOLUTION
   LOOSELY FILL THE COLUMN WITH Cu-Cd
3. ADJUST FLOW RATE
4. STORAGE OF COLUMN
   COVER Cu-Cd GRANULES WITH NH_4CL-EDTA SOLUTION
5. ACTIVATE THE COLUMN OF Cu-Cd
   USE 1.00 mg/l NITRATE SOLUTION
   USE NH_4CL - EDTA SOLUTION

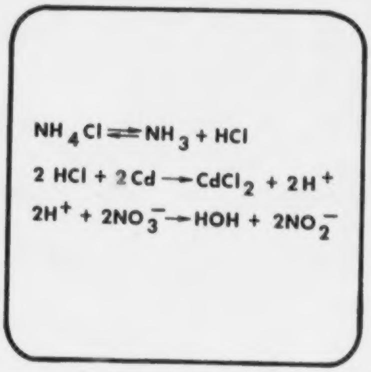
SLIDE 13


Nitrite Plus Nitrate Cadmium Reduction

Removal of interferences to column function

1. Prior to analysis
   Filtration removes suspended solids
   Extraction removes oil and grease
2. During analysis
   Complex metals with EDTA

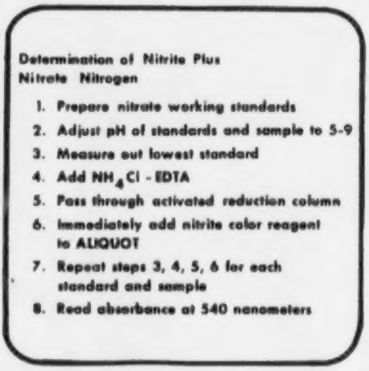
SLIDE 14


$NH_4Cl \rightleftharpoons NH_3 + HCl$

$2\,HCl + 2\,Cd \rightarrow CdCl_2 + 2H^+$

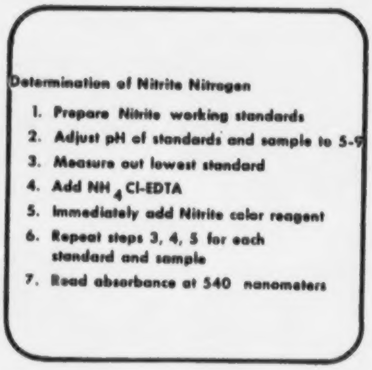
$2H^+ + 2NO_3^- \rightarrow HOH + 2NO_2^-$

SLIDE 15


Determination of Nitrite Plus Nitrate Nitrogen

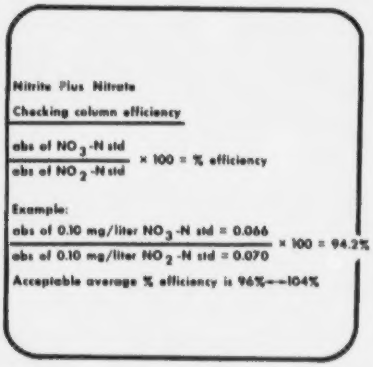
1. Prepare nitrate working standards
2. Adjust pH of standards and sample to 5-9
3. Measure out lowest standard
4. Add NH_4Cl - EDTA
5. Pass through activated reduction column
6. Immediately add nitrite color reagent to ALIQUOT
7. Repeat steps 3, 4, 5, 6 for each standard and sample
8. Read absorbance at 540 nanometers

SLIDE 16


Determination of Nitrite Nitrogen

1. Prepare Nitrite working standards
2. Adjust pH of standards and sample to 5-9
3. Measure out lowest standard
4. Add NH_4Cl-EDTA
5. Immediately add Nitrite color reagent
6. Repeat steps 3, 4, 5 for each standard and sample
7. Read absorbance at 540 nanometers

SLIDE 17


Nitrite Plus Nitrate

Checking column efficiency

$$\frac{\text{abs of } NO_3\text{-N std}}{\text{abs of } NO_2\text{-N std}} \times 100 = \%\ \text{efficiency}$$

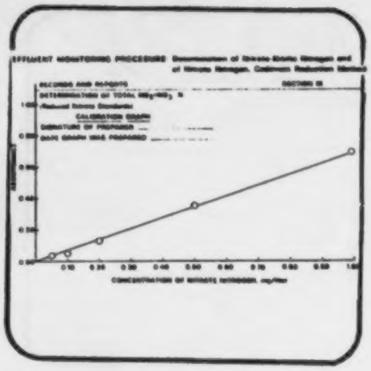
Example:

$$\frac{\text{abs of 0.10 mg/liter } NO_3\text{-N std} = 0.066}{\text{abs of 0.10 mg/liter } NO_2\text{-N std} = 0.070} \times 100 = 94.2\%$$

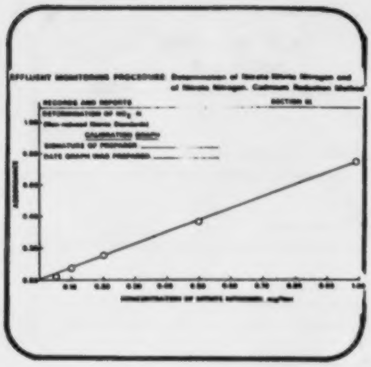
Acceptable average % efficiency is 96%---104%

SLIDE 18

SERIES X:23 NITRITE/NITRATE NITROGEN



SLIDE 19



SLIDE 20

GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Solids

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 190 minutes (15+10+60+45+30+10+10+10)
Note: This is time per one station. See VIII. B.2. for estimating weighing times according to number of stations and balances.

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of equations for inorganic reactions

B. Ability to use analytical balance

C. Ability to use graduates and filtration apparatus

D. Ability to perform calculations involving decimals

E. Completion of the lessons (or equivalent):

1. Compliance Methodology

2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent information about the procedures to do five gravimetric determinations of residues. He/she will use procedures in the training manual (see VII. A. 1. b. and c.) to determine total residue in a municipal wastewater treatment plant influent and nonfilterable (suspended) residue for a plant influent (membrane filter apparatus) and for a plant effluent (Gooch crucible), checking for constant weight after a second drying and reporting results.

B. Conditions: He/she will be given the training manual, 105 minutes of instruction, 75 minutes laboratory time, all necessary equipment, reagents and samples, and 10 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor and obtain results for each sample within ± 10% of the class average if constant weight after drying was achieved during the alloted time.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:
    a. "Testing for Solids in Water Samples"
    b. "Laboratory Procedure for Total Solids" according to 1979 EPA Methods for Chemical Analysis, p. 160.3-1
    c. "Laboratory Procedure for Suspended (Non-Filterable) Solids" according to 1979 EPA Methods for Chemical Analysis, p. 160.2-1
2. Thirty-two slides, X-28: Solids (See XI Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.
2. Clean and permanently mark identification on evaporating dishes and on Gooch crucibles to be used by the class. Weigh and record rough weights for each on a sheet(s) to post by balances.
3. Fire the evaporating dishes at 550 ± 50°C for 1 hour in a muffle furnace, partially cool and put in a desiccator, ready for weighing by class.
4. Put a glass fiber filter disc into each Gooch crucible and wash and dry each according to the procedure in the training manual. Discard any non-usable discs. Prepare one for each station plus a few extra. Place in a desiccator ready for weighing by class.
5. Mark identification numbers on planchets (or watch glasses). Use a membrane filter apparatus and wash glass fiber filter discs. Place each on a marked planchet (or watch glass) and store in a desiccator, ready for weighing by class. Prepare a few extras.
6. Check ovens for use at 98°C to evaporate total residue samples and at 103-105°C to dry total and suspended residue samples.
7. Arrange to get influent and effluent samples from a municipal wastewater treatment plant, as listed in X. below.
8. Review the laboratory procedures by performing the student assignments using the outlines in the training manual.
9. Review the topic outline in the training manual and the visual materials. See XI below.

10. Prepare the lesson using the sequencing below or your own organization of the material.

11. Assemble items for classroom briefing on initial weighings: Gooch crucible containing disc, membrane filter funnel and support and corresponding disc, watch glass for disc, forceps, 100-150 ml evaporating dish.

12. Distribute supplies and reagents to laboratory stations.

13. OPTIONAL: You might want to plan to demonstrate the Imhoff cone method to determine settleable solids if your participants need this information. You will need 1 liter of sample containing settleable solids, an Imhoff cone in a support, and a timer. The demonstration can be done during a laboratory session.

B. Sequencing: Slides are series X-28: Solids

1. Classroom - Briefing (15 minutes) on weighings to be done during combined laboratory-lunch break. This plan works well when the participant to balance ratio is large.

   a. Explain why this scheduling. Lectures will be after lunch.

   b. Use outline on Total Solids. Show evaporating dish; give overview of test; III. A. B. and C. are done; assign D.; record weight on outline sheet in manual. Give location of desiccators and balances.

   c. Use outline on Suspended Non-Filterable Solids. Show membrane filter funnel and support and corresponding disc on a watch glass; note its use for influents; disc must be carefully transferred to balance pan with forceps and weighed. Show Gooch crucible containing disc; it stays in crucible for weighings; note its use for effluents; use tissue when handling. III. A. B. C. D. E. are done. Assign F. Desiccator and balance locations are the same as above. Stress that the constant weight check should be done for "real" tests, but in the interest of time they will omit this part of III F.

   d. A data sheet for Suspended and Non-Filterable solids is in the manual. They mark sample at top of column as "INF-MF" and "EFF-G" to ensure correct apparatus is used for the two samples. Note blank (1) for identification record on filters, and blank (14) for initial weight and the referenced* footnote. (Save any other discussion of the data sheet for lecture session). They should use same balance for all subsequent weighings. Rough weights of crucibles are posted by balances.

   e. Summarize - Three weighings are to be accomplished and recorded: an evaporating dish, a 4.7 cm disc and a 2.2 cm disc in a Gooch crucible.

2. Laboratory - 10 minutes per 3 weighings. Time allotment on agenda depends on number of stations and number of available balances. (Ex. 12 stations and 4 balances required 30 minutes). One way

to avoid back-ups is to schedule half of class first while others have a break or go to lunch, then first has a break or is at lunch while others use balances.

3. Classroom - Introduction - 8 minutes
   Participants should use the training outline, "Testing for Solids in Water Samples" as a reference for the following discussions.

   a. Section II Solids and Water Quality - Overview of why tests are done.

   b. Section IV lists NPDES Solids Tests

   c. Section V - accuracy of tests and term, "residue"

   d. Section VI - All the tests are gravimetric determinations of residues remaining after test treatments. Settleable solids may also be reported in terms of volume. As required by the class, highlight information in this section. Do include B, Drying Temperature, to emphasize the differences and why they were chosen.

   e. Section III, Sample Handling - Slide 1

4. Classroom - Total Residue - 10 minutes
   Participants should use outline Section VII, "Total Residue"

   a. Definition from view of components - Slide 2
      Operational definition is in manual,
      Briefly note manual sections on why test is done.

   b. Stepwise procedure - Slides 3,4

      1) Composition of dish

      2) Sample volume limits - in manual

      3) Steps 1 and 2 done. They did 3 and will do 4 through 7 in laboratory

      4) Sources of error - see manual

      5) Constant weight limits - in manual

5. Classroom - Total Dissolved Residue - 10 minutes
   Participants should use outline Section VIII, "Total Dissolved (Filterable) Residue."

   a. Terms filterable and non-filterable illustrated - Slide 5

   b. Definition filterable (dissolved) - Slide 6

   c. Effects - Slide 7
      WWTP significance is in manual

   d. Stepwise Procedure - Slides 8, 9, 10, 11

      1) Sample volume limits - in manual

        2) Constant weight limits - in manual

    e. Interferences - Sources of Error - Slide 12

6. Classroom - Total Suspended Residue (Nonfilterable Solids) - 11 minutes
   Participants should use outline Section IX, "Total Suspended (Non-Filterable Residue)".

    a. Definition - Slide 13

    b. Effects - Slide 14
       WWTP significance is in manual

    c. NPDES regulations - Slide 15

        1) This is in the manual

        2) May be different for lagoons - this is in the manual

    d. Stepwise Procedure - Slides 16, 17

        1) Can use crucible (effluents) or membrane filter support with coarse fritted disc (influents) for the disc.

        2) Steps 1 and 2 done, they did step 3 and will do 4 through 6 in the laboratory for an effluent and an influent sample

        3) Sample volumes - in manual

        4) Sources of error - in manual

        5) Constant weight limits - in manual

7. Classroom - Settleable Residue - 10 minutes
   Participants should use outline Section X, 'Settleable Residue".

    a. Definition, relationship to suspended - Slide 18

    b. Effects - Slide 19

        1) Blanket effect - affects biological activity which affects CO_2 content and, thus, pH. pH affects adsorption/release of organics and heavy metals.

        2) WWTP significance is in manual.

    c. Procedure by weight - Slide 20

    d. Procedure by volume - Slide 21

    e. Interferences and sources of error for both procedures are in manual.

8. Classroom - Conclusion - 3 minutes

   a. Repeat temperatures for drying

   b. Remind about the constant weight condition - may need several weighings

   c. Note accuracy variables, especially representative sample. It is very important to have consistent technique.

   d. Suspended solids quality control samples are available through EPA Regional Quality Assurance Coordinators.

9. Classroom - Lab Briefing - 8 minutes

   a. Total - Influent sample - In the training manual procedure for "Total Solids", do III E. F. G. H. For I, put the dish in a 98°C oven. The Staff will do J. After cooling, the participants will do K and return the dish to a 103 - 105°C oven for a second drying cycle and weighing.

   b. Suspended (Non-Filterable) - In the training manual for "Suspended (Non-Filterable) Solids", do III G through R for an influent sample using a large disc in a membrane filter funnel. Do G through Q, then S for an effluent sample using a disc in a Gooch crucible. The staff will remove items from the oven. After cooling, participants will weigh the items and return them to a 103-105°C oven for a second drying cycle and weighing. The data sheet has blanks for all the data.

10. Break - 10 minutes

11. Laboratory - 45 minutes
    Student performance of the assignment up toputing items in the ovens for the first time.

12. Classroom - Volatile Solids - 30 minutes
    Participants should use outline Section XI, "Total Volatile Residue".

    a. Introduction, title - Slide 22

    b. Definition and relationship to other solids (residues) - Slide 23

    c. Why test is done - Slides 24, 25
       Effects and WWTP significance is in manual

    d. Steps of test - Slides 26, 27

    e. Define fixed residue - Slide 28

    f. Preparation of dish or crucible - Slide 29

    g. Negative errors in the determination, examples - Slides 30,31

    h. General sources of error - Slide 32 (last slide)
       Also are in manual

i. Conclusion

13. Staff - For total residue, increase temperature in oven from 98°C to 103-105°C after samples evaporate. For all residues, tranfer dishes, discs and supports to desiccators after first drying so they are at room temperature for first weighing.

14. Laboratory-First weighing of two Non-filterable (suspended) residues - 7 minutes (See 9 above). Put discs back in oven.

    First weighing of one Total residue - 3 minutes (See 9 above). Put dish back in oven.

15. Staff - Later, remove discs and supports from oven and put into desiccators so they are at room temperature for second weighing.

16. Laboratory - Second weighing of all three residues - 10 minutes

17. Class Discussion - 10 minutes

    a. Review the constant weight check limit for each test. Have participants compare the two weights they obtained for each of the three tests and calculate the differences.

    b. Participants should post the differences on the board.

    c. Discuss the results posted for the constant weight check.

    d. Participants should use the calculation formulas (IV in laboratory procedures or, for suspended residue, a stepwise calculation is on the data sheet). They should post results on the board. Discuss the results. Check on very low results, especially for the two suspended (non-filterable) tests. EPA recommends having at least 0.47 mg residue on a 2.2 cm disc and 1.0 mg on a 4.7 cm disc. Participants should check their residue weights accordingly.

    e. Give concluding cautions on techniques involved, e.g. handling the discs, finger prints on dish, etc..

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: One total residue test and one non-filterable (suspended) residue test on an influent sample
One non-filterable (suspended) residue test on an effluent sample

A. For each station: (no more than two persons per station):

1. Enough desiccator space for one 150 ml evaporating dish, one Gooch crucible, and one small planchet (or watch glass). Desiccant should be usable.

2. One 150 ml evaporating dish with a permanent identification mark on it, stored in a desiccator, ready for weighing.

3. One glass fiber filter disc in a Gooch crucible (permanent identification mark), washed, dried and in a desiccator, ready for weighing.

4. One glass fiber filter disc to fit a membrane filter apparatus, washed, dried and stored on a marked planchet (or watch glass) in a desiccator, ready for weighing.
5. One 25 or 50 ml graduated cylinder
6. One 100 ml graduated cylinder
7. One suction flask with hose and pinch clamp
8. One membrane filter holder assembly with rubber stopper to fit neck of suction flask
9. One pair blunt-tipped forceps
10. One rubber adapter to hold crucible in neck of suction flask
11. One pair tongs
12. One vacuum source or pump drawing 15 inches of mercury
13. One squeeze bottle of distilled water
14. One marking pencil
15. One apron for each student
16. One pair safety glasses for each student

B. Shared:

1. Desiccators with usable desiccant. See A.1. above for space requirement per station.
2. Box small tissues by each desiccator
3. Balances, analytical - One per 3 stations
4. Box small tissues by each balance
5. Drying oven or steam bath for use at 98°C and to accommodate one 150 ml evaporating dish per station
6. Oven for use at 103-105°C and to accommodate three items per station: one 150 ml evaporating dish, one Gooch crucible, and one small planchet (or watch glass).

X. IPW REAGENT REQUIREMENTS:

A. Minimum amounts per station (no more than two persons per station):

1. Enough plant influent sample to yield 25 to 200 mg total or suspended residue
2. Enough plant effluent sample to yield 25 to 200 mg suspended residue

3. EPA-EMSL, Cincinnati, Ohio 45268 supplies free analytical quality control samples containing known amounts of suspended solids. This might be an alternative if plant samples are not available.

B. Obtain excess of each sample to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory sessions.

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Thirty-two slides, X-28: Solids - See next pages.

SERIES X-28: SOLIDS

SAMPLE HANDLING

- RESISTANT GLASS BOTTLES OR PLASTIC CONTAINERS
- NO PRESERVATIVE
- ANALYZE AS SOON AS POSSIBLE
- REMOVE LARGE PARTICULATES (LEAVES, TWIGS, ETC.)

SLIDE 1

TOTAL SOLIDS

THE SUM OF THE HOMOGENEOUS

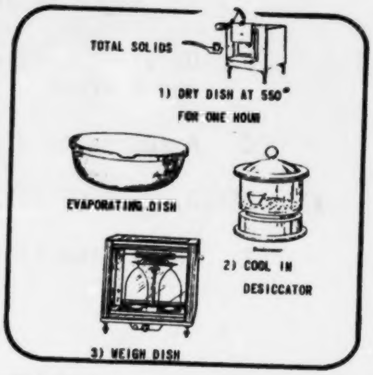
SUSPENDED

AND

DISSOLVED

MATERIALS IN A SAMPLE

SLIDE 2



TOTAL SOLIDS

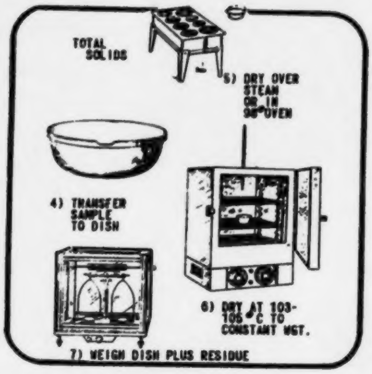
1) DRY DISH AT 550° FOR ONE HOUR

EVAPORATING DISH

2) COOL IN DESICCATOR

3) WEIGH DISH

SLIDE 3



TOTAL SOLIDS

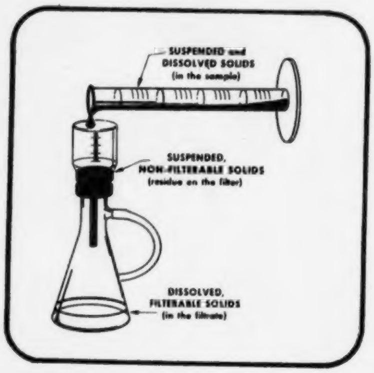
4) TRANSFER SAMPLE TO DISH

5) DRY OVER STEAM OR IN 98°OVEN

6) DRY AT 103-105 °C TO CONSTANT WGT.

7) WEIGH DISH PLUS RESIDUE

SLIDE 4



SUSPENDED and DISSOLVED SOLIDS (in the sample)

SUSPENDED, NON-FILTERABLE SOLIDS (residue on the filter)

DISSOLVED, FILTERABLE SOLIDS (in the filtrate)

SLIDE 5

FILTERABLE SOLIDS

THOSE SOLIDS

PASSING THROUGH A STANDARD GLASS FIBER FILTER

AND

DRIED TO CONSTANT WEIGHT AT 180 DEGREES C

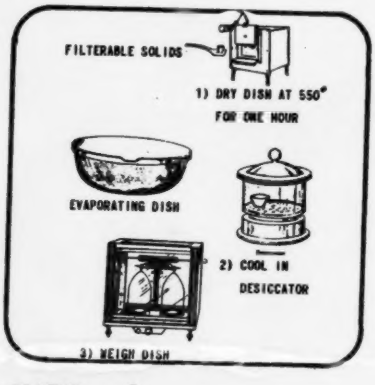
SLIDE 6

FILTERABLE SOLIDS

EFFECTS

1) PUBLIC WATER SUPPLIES
    - TASTE, PHYSIOLOGICAL
    - TOXICITY, CORROSION
2) IRRIGATION
    - NUTRIENT BALANCE
    - TOXICITY, OSMOTIC PRESSURE
3) LIVESTOCK
    - TASTE, PHYSIOLOGICAL
    - TOXICITY
4) AQUATIC LIFE
    - NUTRIENTS
    - TOXICITY, OSMOTIC PRESSURE

SLIDE 7



FILTERABLE SOLIDS

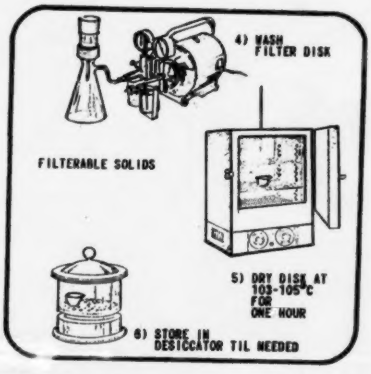
1) DRY DISH AT 550° FOR ONE HOUR

EVAPORATING DISH

2) COOL IN DESICCATOR

3) WEIGH DISH

SLIDE 8



FILTERABLE SOLIDS

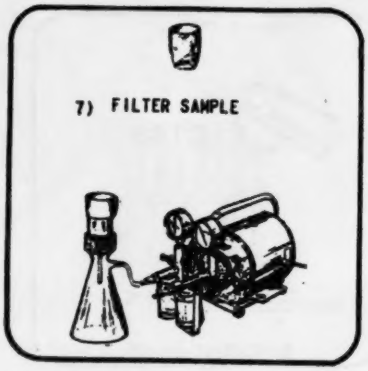
4) WASH FILTER DISK

5) DRY DISK AT 103-105°C FOR ONE HOUR

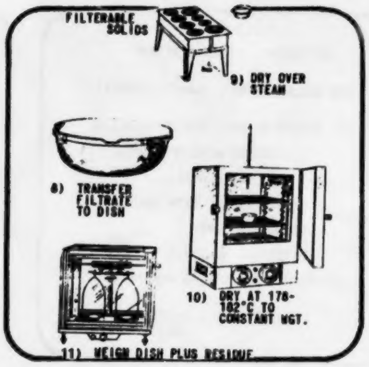
6) STORE IN DESICCATOR TIL NEEDED

SLIDE 9

SERIES X-28: SOLIDS


7) FILTER SAMPLE

SLIDE 10


FILTERABLE SOLIDS

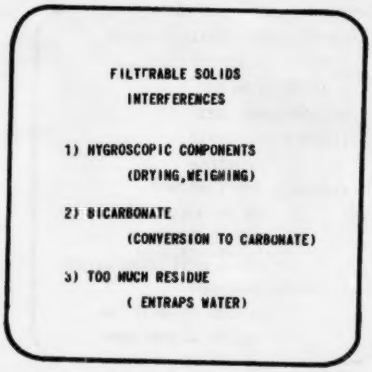
8) TRANSFER FILTRATE TO DISH

9) DRY OVER STEAM

10) DRY AT 178-182°C TO CONSTANT WGT.

11) WEIGH DISH PLUS RESIDUE

SLIDE 11


FILTERABLE SOLIDS

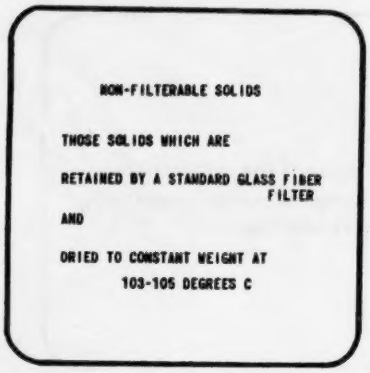
INTERFERENCES

1) HYGROSCOPIC COMPONENTS
(DRYING,WEIGHING)

2) BICARBONATE
(CONVERSION TO CARBONATE)

3) TOO MUCH RESIDUE
(ENTRAPS WATER)

SLIDE 12


NON-FILTERABLE SOLIDS

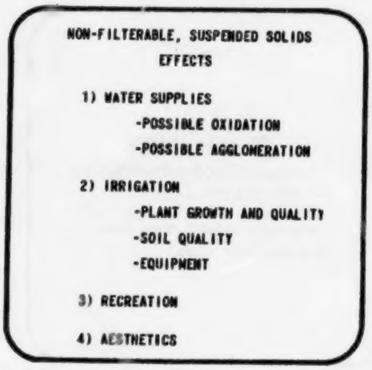
THOSE SOLIDS WHICH ARE

RETAINED BY A STANDARD GLASS FIBER FILTER

AND

DRIED TO CONSTANT WEIGHT AT
103-105 DEGREES C

SLIDE 13


NON-FILTERABLE, SUSPENDED SOLIDS

EFFECTS

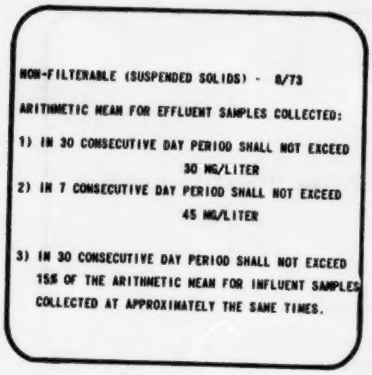
1) WATER SUPPLIES
- -POSSIBLE OXIDATION
- -POSSIBLE AGGLOMERATION

2) IRRIGATION
- -PLANT GROWTH AND QUALITY
- -SOIL QUALITY
- -EQUIPMENT

3) RECREATION

4) AESTHETICS

SLIDE 14


NON-FILTERABLE (SUSPENDED SOLIDS) - 8/73

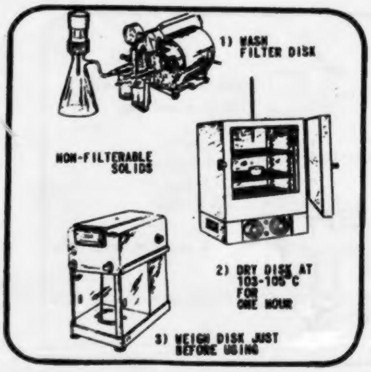
ARITHMETIC MEAN FOR EFFLUENT SAMPLES COLLECTED:

1) IN 30 CONSECUTIVE DAY PERIOD SHALL NOT EXCEED
30 MG/LITER

2) IN 7 CONSECUTIVE DAY PERIOD SHALL NOT EXCEED
45 MG/LITER

3) IN 30 CONSECUTIVE DAY PERIOD SHALL NOT EXCEED
15% OF THE ARITHMETIC MEAN FOR INFLUENT SAMPLES
COLLECTED AT APPROXIMATELY THE SAME TIMES.

SLIDE 15

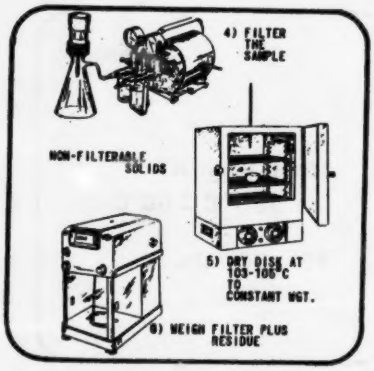

1) WASH FILTER DISK

NON-FILTERABLE SOLIDS

2) DRY DISK AT 103-105°C FOR ONE HOUR

3) WEIGH DISK JUST BEFORE USING

SLIDE 16

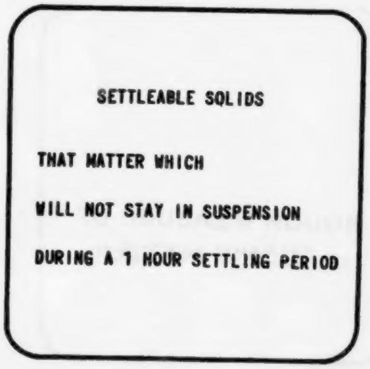

4) FILTER THE SAMPLE

NON-FILTERABLE SOLIDS

5) DRY DISK AT 103-105°C TO CONSTANT WGT.

6) WEIGH FILTER PLUS RESIDUE

SLIDE 17


SETTLEABLE SOLIDS

THAT MATTER WHICH

WILL NOT STAY IN SUSPENSION

DURING A 1 HOUR SETTLING PERIOD

SLIDE 18

SERIES X-28: SOLIDS

NON-FILTERABLE, SETTLEABLE SOLIDS

EFFECTS

1) AESTHETIC VALUES
2) RECREATIONAL USES
3) MECHANICAL DAMAGES
    - NAVIGATION
4) BLANKET EFFECT DESTROYS
    - BOTTOM FAUNA / BIOTA
    - SPAWNING GROUNDS
    - FISH AND SHELLFISH
    - ORGANIC WASTE DECOMPOSITION
5) ORGANIC COMPONENTS
    - DEPLETE BOTTOM OXYGEN
    - PRODUCE NOXIOUS GASES

SLIDE 19

SETTLEABLE SOLIDS - WEIGHT

(1) DETERMINE MG/L NON-FILTERABLES

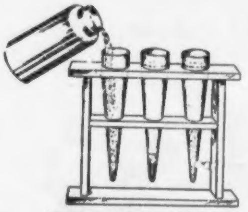
(2) DETERMINE MG/L NON-SETTLEABLES
    - 1 LITER OR MORE OF SAMPLE
    - 1 HOUR QUIESCENCE
    - SIPHON 250 ML FROM CENTER OF SUPERNATANT
    - DETERMINE MG/L NON-FILTERABLES

(3) REPORT RESULTS IN MG/LITER

(1) MINUS (2) = SETTLEABLE SOLIDS

SLIDE 20

IMHOFF CONE



After filling the Imhoff cone to the 1-l mark (left), allow to settle for 45 min (center), then stir gently (right) and settle for 15 min more.

SETTLEABLE SOLIDS

SLIDE 21

TOTAL VOLATILE RESIDUE

mg/l

SLIDE 22

VOLATILE SOLIDS

THOSE SOLIDS COMBUSTIBLE AT 550 DEGREES C

(May apply to total, filterable, or non-filterable solids)

SLIDE 23

REASONS FOR DETERMINATION

- WATER QUALITY REQUIREMENT
- PLANT CONTROL

SLIDE 24

ROUGH MEASURE OF ORGANIC MATTER

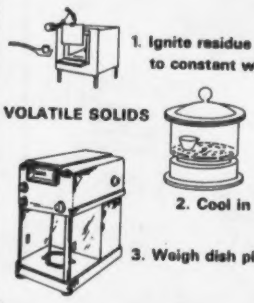
SLIDE 25

IGNITION AT

550°C ± 50°C

15 - 20 min.

SLIDE 26



VOLATILE SOLIDS

1. Ignite residue at 550°C to constant wgt.
2. Cool in desiccator
3. Weigh dish plus residue

SLIDE 27

SERIES X-28: SOLIDS

FIXED RESIDUE

SLIDE 28

550°C ± 50°C PRE-IGNITION OF DISH OR CRUCIBLE

SLIDE 29

$MgCO_3 \xrightarrow{350°C} MgO + CO_2\uparrow$

SLIDE 30

$(NH_4)_2CO_3 \rightarrow 2NH_3\uparrow + H_2O\uparrow + CO_2\uparrow$

SLIDE 31

SOURCES OF ERROR

- Uncontrolled temperature

  $CaCO_3 \xrightarrow{825°C} CaO + CO_2\uparrow$
  (major)
- Volatiles loss before ignition
- Incomplete organics oxidation
- Loss of water of crystallization
- Handling of dish

SLIDE 32

GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Elements of a Quality Assurance Program

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 75 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know and practice requirements for quality assurance when conducting analytical procedures and reporting results.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of quantitative analyses

B. Familiarity with equipment and supplies normally found in a water quality chemistry laboratory

C. At least six months experience in chemical laboratory work

D. Completion of the lessons (or equivalent):

1. Sample Handling
2. Compliance Methodology
3. Analytical Techniques
4. Volumetric Analysis
5. Precision
6. Accuracy
7. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will be able to utilize the given outline (see VII. 1. a.) of activities required to effectively fulfill the demands of a quality assurance program for a chemistry laboratory. (VIII. B, Sequencing, in this IPW lists the divisions of these activities)

B. Conditions: He/she will be given the training manual, handouts and 75 minutes of discussion.

C. Accepted Performance: He/she must recall from memory at least 70% of the items pertaining to the objective in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:
    a. "Elements of a Quality Assurance Program" which is primarily an outline of the information in resource 3. below.
    b. "Methodology for Chemical Analysis of Water and Wastewater" which defines the principles utilized in recognized methodology.
    c. "Use of a Spectrophotometer" has a section (IV) on calibration standards.
2. Five slides, X-26: Quality Assurance (See XI Description of Visual Materials)
3. Resource: EPA "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", Environmental Monitoring & Support Laboratory (EMSL), Cincinnati, Ohio 45268. Copies can be ordered from EMSL: 513 684-7301.
4. Two Handouts - To ensure that current information is distributed, request this information from U.S. EPA, EMSL - Quality Assurance Branch, Cincinnati, Ohio 45268:
    a. The current list of quality control samples available from EPA through the EPA Regional Quality Assurance Coordinators.
    b. (Optional): Roster of EPA Regional Quality Assurance Coordinators if students are from several regions
5. One Handout - Sheet with examples of significant figures and basics on "rounding off" and calculating rules. A copy is at the end of this IPW.

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review visual materials and available handouts.
2. Prepare the lesson using the Sequencing below or your own organization of the material.
3. Duplicate copies of handout materials (See VII. A. 4.) for each student. Assemble in packets.
4. Assemble demonstration items: volumetric flask, volumetric pipet and small tissues for demonstration of glassware markings and swiping technique for pipets.

B. Sequencing:

Use indicated sections of topic outline in the training manual to discuss these topics unless otherwise noted.
Slides are series X-26: Quality Assurance

1. Introduction - I. A. and B. Note that there are divisions in the outline with details on the 5 elements in I. C. This is primarily an outline of highlights of reference 1. Then briefly cover I.D. and I.E.

2. II. Sample - This was considered in detail in prior lesson, "Sample Handling"

3. III. Recognized Methodology - This was considered in detail in prior lesson, "Compliance Methodology."

    a. D. - definitions of commonly-used types of methodology are in course manual outline, "Methodology for Chemical Analysis of Water and Wastewater."

    b. F. Stress strict adherence to reference source when using recognized procedures.

4. IV. Services, Instruments, Supplies - Give highlights. Some notes:

    a. B. 2. g. - blank plus one, preferably two, standard(s) may be used to check a curve for some methods, rather than running a complete set of standards each run.

    b. C. 2. the four functions: a, b, c, d

    c. C. 2. d. Show volumetric flask, volumetric pipet and measuring pipet to demonstrate location of glassware information and also TD and TC terms - Also use pipet and tissue to demonstrate swiping excess off outside of pipet before final reading.

    d. D. 2. General guides are here (a, b, c, d) for their future reference.

    e. D. 5. Standard solutions are in lesson, "Volumetric Analysis."

    f. D. 5. a. 6) Outline "Use of a Spectrophotometer" has Section on Preparation of Calibration Standards, IV. B. Discuss stability information there.

    g. D. 5. c. This is especially important for "Compliance Methodology."

5. V. Quality Analytical Performance

    a. A. Summarize

    b. B. and C. Recall from prior lessons, "Precision" and "Accuracy"

c. D. Note 1, 2, 3, 4

d. E. Present items 1, 2, 3, 4, 5

e. Slides of normal curve and control charts using some of the statistics presented in lessons on precision and accuracy:

1) Normal curve - Relate midline, 2 s and 3 s limits to control chart central line, warning and control limits. Slide 1.

2) Precision Chart - R as estimate of s using duplicates. Plot succeeding R of duplicates. Slide 2 (This approach is recommended in the 1979 EPA Handbook for Analytical Quality Control).

3) Precision Chart - $\overline{X}$ and s, using one check standard. Plot succeeding results. Slide 3.

4) Accuracy Chart - $\overline{X}$ and s of % recoveries using spiked samples. Plot succeeding % recoveries of spikes. Slide 4 (This approach is recommended in the 1979 EPA "Handbook for Analytical Quality Control".)

5) Accuracy Chart - $\overline{X}$ and s of % bias using spiked samples. Plot succeeding % bias (% relative error). Slide 5

f. F. Interlaboratory Checks on Performance

1) F. 1. Hand out the list of EPA quality control samples currently available through EPA Regional Quality Assurance Coordinators. Give the Participants the telephone and address information for their Regional Coordinator.

2) F, 2, 3, 4, 5 - Note these checks

6. VI. Data Handling and Reporting

a. A. 1, 2, 3

b. A. 4 Hand out and discuss the sheet on significant figures. Relate it to course methodology. Note reference for more detail.

c. D. 1. Note current Federal Register regulations regarding storage of data.

7. VII. Safety Considerations - Note highlights. This was considered in detail in a previous lesson. Encourage appointment of Safety Officer.

8. VIII. EPA Regional Quality Assurance Coordinators.

a. Encourage contact with EPA coordinator.

9. References - Note the list

    10. IX. Summary

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For each student:

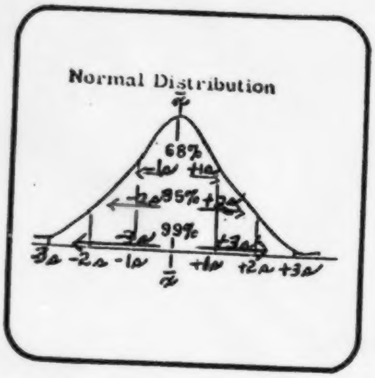
1. Copies of handout materials

X. IPW REAGENT REQUIREMENTS:

A. None

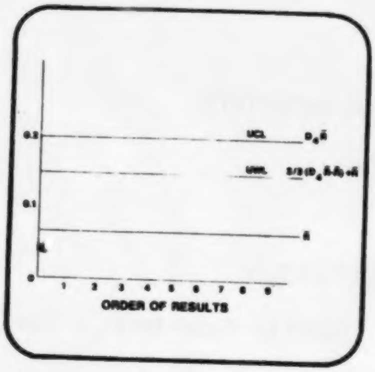
XI. DESCRIPTION OF VISUAL MATERIALS:

A. Five slides, X-26: Quality Assurance - See next page.

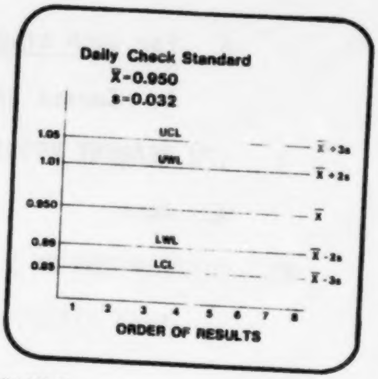


Normal Distribution

SLIDE 1

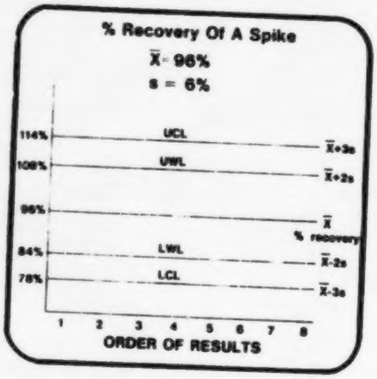


SLIDE 2



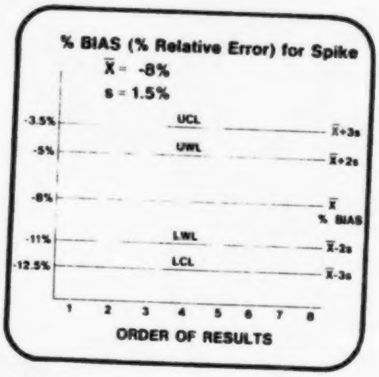
Daily Check Standard
$\bar{X}$=0.950
s=0.032

SLIDE 3



% Recovery Of A Spike
$\bar{X}$= 96%
s = 6%

SLIDE 4



% BIAS (% Relative Error) for Spike
$\bar{X}$= -8%
s = 1.5%

SLIDE 5

SIGNIFICANT FIGURES - EXAMPLES

32.2 has 3 significant figures
32.20 has 4 significant figures
0.0014 has 2 significant figures (zeros used to fix decimal location)
0.1400 has 4 significant figures
0.14 has 2 significant figures (the zero indicates no whole number)

*** *** *** ***

1. Retain as many significant figures in a result or in laboratory data as will give only one uncertain figure. (Possibly, in very accurate work involving lengthy computations, two uncertain figures may sometimes be retained.)
2. When rounding off, if the rejected figure is 0, 1, 2, 3, or 4, just let the remaining significant figure stand as is. If the rejected figure is 5, round the remaining figure to the nearest even number: 2.25 becomes 2.2 and 2.35 becomes 2.4. If the rejected figure is 6, 7, 8, or 9, increase the remaining significant figure by one unit.
3. As a practical operating rule for multiplication or division, keep as many significant figures in an answer as were contained in the factor having the least number of significant figures. The answer should not "suffer" if the other factors are limited to a minimum of one more significant figure than that least number of significant figures during the calculation.
4. For addition or subtraction, the number with the fewest decimal places determines the number of decimal places retained. However, no one number involved in the calculation operation limits the number of significant figures in the result.

Source: Standard Methods for the Examination of Water and Wastewater, 14th edition, APHA-AWWA-WPCF, 1975, pp. 17-20.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Turbidity

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 95 minutes (45+40+10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Ability to correctly use volumetric glassware

B. Completion of the lesson (or equivalent):

1. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent facts about turbidity and procedures to determine turbidity (see VIII. B.). He/she will dilute a stock solution to make standards (see VIII. B. 2.), standardize a turbidimeter ensuring that the cell will be consistently oriented in the meter, measure the turbidity of a sample using the procedure in the training manual (see VII. A. 1. b.), report results and participate in a class discussion of the topic and laboratory procedures.

B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 40 minutes laboratory time, all necessary equipment, a stock solution and sample, and 10 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor and obtain results for the sample within ± 10% of the class average (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the training manual:

a. "Turbidity"

b. "Calibration and Use of a Turbidimeter (Nephelometer)" according to 1979 EPA Methods for Chemical Analysis, 180.1-1

2. Ten slides, X-30: Turbidity (See XI Description of Visual Materials).

B. Suggested Media:

   1. None

VIII. INSTRUCTIONAL APPROACH:

   A. Preparation for Instruction:

      1. Check equipment and supplies. See IX below.
      2. Prepare all necessary reagents and sample as in X below.
      3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.
      4. Review the topic outline in the training manual and the visual materials. See XI below.
      5. Prepare the lesson using the Sequencing below or your own organization of the material.
      6. Check turbidimeters for accurate operation.
      7. Distribute supplies and reagents to laboratory stations.

   B. Sequencing: Slides are series X-30: Turbidity

      1. Classroom instruction on turbidity - 45 minutes

         a. Introduction

            1. Cause - Slide 1
            2. Why measure turbidity - Slide 2
            3. Sample handling - preservation and holding time - Slide 3 (For compliance monitoring, maximum holding times proposed during 1979 were 24 hours for drinking water samples and 48 hours for NPDES samples. Consult current federal registers for current status).
            4. Original Jackson Candle Turbidimeter - Slide 4

         b. Regulations - NPDES and Water Supply both require nephelometers.

         c. Acceptable instrumentation

            1) Nephelometry - Slides 5, 6
            2) Formazin Stock Solution - Slides 7, 8, 9
               Note: A final decision on use of styrene divinylbenzene polymer standards for drinking water calibration standards is to be made after 10/27/80. Check on the status of approval before presenting this.
            3) Interferences - Slide 10

         d. Summary

2. Laboratory - 40 minutes

   a. Briefing - Participants use training manual "Calibration and Use ..." outline, III. A., 1 through 17 to use the provided 400 NTU standard for preparing three (40, 4 and 0.4 NTU) standards for the DRT instrument or four (40, 4, 0.4 and 0.04 NTU) standards for the Hach 2100 A instrument, standardizing the instrument, then determining turbidity on the provided sample and on tap water.

   b. Student performance of the assignment

3. Class Discussion - 10 minutes

   a. Participants report results.

   b. Cover any items from laboratory sessions of general nature.

   c. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Dilute the stock solution to make one calibration standard for each range of the instrument to be used, e.g., 40, 4, 0.4, 0.04.

Test two samples (tap water plus 1 synthetic).

To minimize the number of turbidimeters required, the laboratory could be conducted twice with half the class doing another procedure requiring about the same time, e.g., Specific Conductance.

A. For each station (no more than two persons per station):

   1. One pipet bulb
   2. Three or four 100 ml volumetric flasks with stoppers
   3. Three or four 10 ml volumetric pipets if it contains measurable turbidity
   4. One turbidimeter (nephelometer) with cell.
      CAUTION: To ensure that the cell is oriented consistently during the procedure, the participants should mark the cell and instrument during the calibration procedure.
   5. One squeeze bottle turbidity - free distilled water
   6. One marking pencil
   7. One apron for each student
   8. One pair safety glasses for each student

B. Shared:

   1. None

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 1979 EPA "Methods for Chemical Analysis", 180.1-1

A. Minimum amounts per station (no more than two persons per station):

1. Ten ml 400 NTU stock Formazin suspension

2. About 50 ml sample (It is convenient to make 1 liter of sample by diluting 5 ml of a 40 NTU standard to 1000 ml for a resulting turbidity of 0.2 NTU)

3. Note: Tap water is the second sample. Also, a treatment plant effluent sample could be used if it contains measureable turbidity.

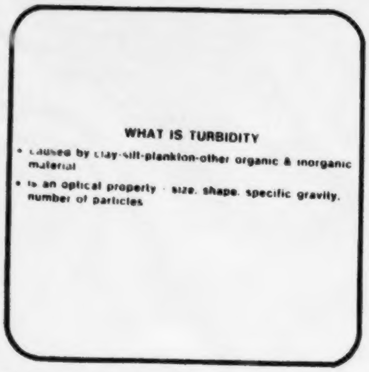
B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of turbidity - free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

SERIES X-30: TURBIDITY

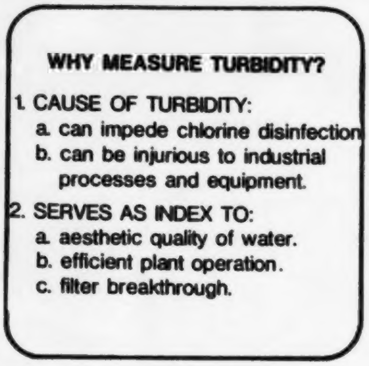
A. Ten slides, X-30: Turbidity



WHAT IS TURBIDITY

- caused by clay-silt-plankton-other organic & inorganic material
- is an optical property - size, shape, specific gravity, number of particles

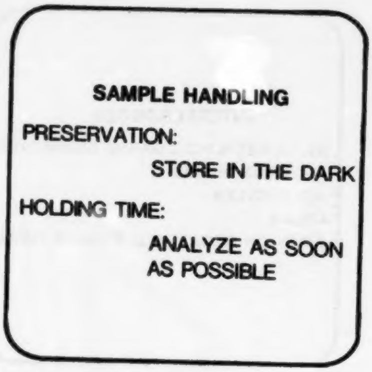
SLIDE 1



WHY MEASURE TURBIDITY?

1. CAUSE OF TURBIDITY:
   a. can impede chlorine disinfection
   b. can be injurious to industrial processes and equipment.
2. SERVES AS INDEX TO:
   a. aesthetic quality of water.
   b. efficient plant operation.
   c. filter breakthrough.

SLIDE 2



SAMPLE HANDLING

PRESERVATION:
STORE IN THE DARK

HOLDING TIME:
ANALYZE AS SOON AS POSSIBLE

SLIDE 3

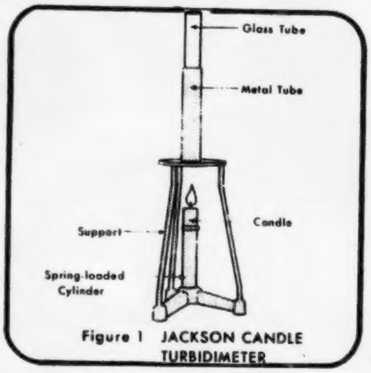
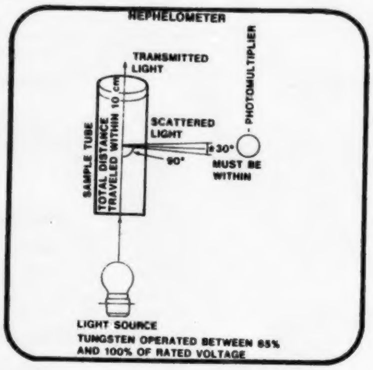

Glass Tube
Metal Tube
Support
Candle
Spring-loaded Cylinder

Figure 1 JACKSON CANDLE TURBIDIMETER

SLIDE 4

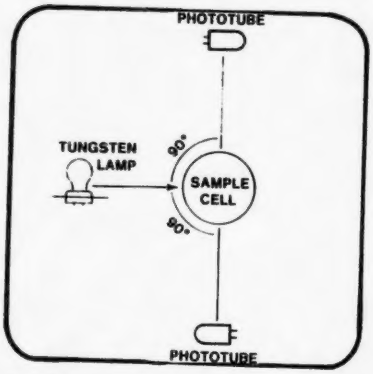


NEPHELOMETER

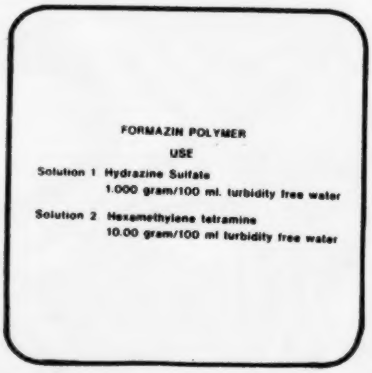
TRANSMITTED LIGHT
PHOTOMULTIPLIER
SAMPLE TUBE
TOTAL DISTANCE TRAVELED WITHIN 10 cm
SCATTERED LIGHT
±30°
90°
MUST BE WITHIN
LIGHT SOURCE

TUNGSTEN OPERATED BETWEEN 85% AND 100% OF RATED VOLTAGE

SLIDE 5


PHOTOTUBE
TUNGSTEN LAMP
90°
SAMPLE CELL
90°
PHOTOTUBE

SLIDE 6



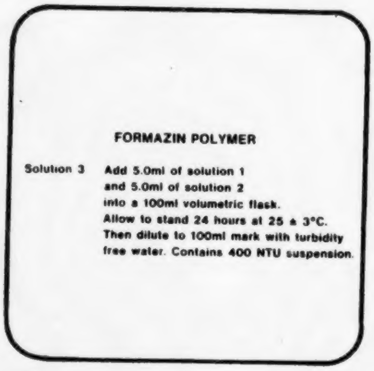
FORMAZIN POLYMER

USE

Solution 1 Hydrazine Sulfate
1.000 gram/100 ml. turbidity free water

Solution 2 Hexamethylene tetramine
10.00 gram/100 ml turbidity free water

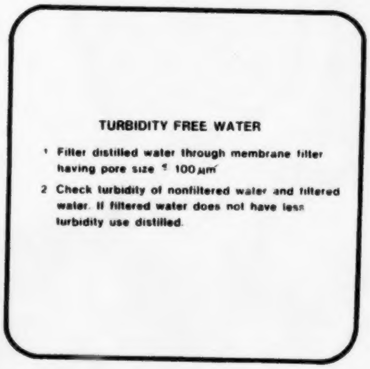
SLIDE 7



FORMAZIN POLYMER

Solution 3 Add 5.0ml of solution 1 and 5.0ml of solution 2 into a 100ml volumetric flask. Allow to stand 24 hours at 25 ± 3°C. Then dilute to 100ml mark with turbidity free water. Contains 400 NTU suspension.

SLIDE 8



TURBIDITY FREE WATER

1. Filter distilled water through membrane filter having pore size ≤ 100 μm
2. Check turbidity of nonfiltered water and filtered water. If filtered water does not have less turbidity use distilled.

SLIDE 9

SERIES X-30: TURBIDITY

INTERFERENCES

RAPID SETTLING COARSE SEDIMENTS
FLOATING DEBRIS
AIR BUBBLES
COLOR
DIRTY OR SCRATCHED SAMPLE TUBES

SLIDE 10

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Specific Conductance

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 105 minutes (45+50+10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of terms "ions", "ionization", "dissociation"

B. Ability to perform calculations involving decimals

C. Completion of the lessons (or equivalent):

1. Volumetric Analysis (terms "acid", "base", "salt", "normality")

2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent facts about specific conductance (see VIII B). He/she will use the procedures in the training manual (see VII A 1 b) to check out a conductivity meter, use it to determine cell constants for three different probes, and determine specific conductance on three samples. He/she will report results and participate in a class discussion of the topic and laboratory procedures.

B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 50 minutes of laboratory time, all necessary equipment, standards and samples and 10 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor and obtain results for each sample within ± 10% of the class average (unless the reason for a lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the training manual:

a. "Specific Conductance"

b. "Calibration and Use of a Conductivity Meter" according to 14th edition Standard Methods, p. 71 and manufacturer's instructions for Beckman's SoluBridge instrument. The outline might serve as a guide if you need to develop laboratory directions for instruments other than this one.

2. Fifteen slides, X-29: Specific Conductance (see XI Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.
2. Prepare all necessary reagents and sample as in X below.
3. Review the laboratory procedure by performing the student assignment using the outline in the training manual. If you have different meter-probe equipment, you will have to revise the training outline or even prepare a new one to fit your needs.
4. Review the topic outline in the training manual and the visual materials. See XI below.
5. Prepare the lesson using the Sequencing below or your own organization of the material.
6. As appropriate, check batteries, conductivity meters and probes for accurate operation.
7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

Slides are series X-29: Specific Conductance

1. Classroom Instruction on Specific Conductance - 45 minutes
   a. NPDES methodology unit, temperature condition, and measurement requirement (slide 1).
   b. Conductivity is an expression of the ability of a water sample to carry an electric current. Discuss the processes resulting in ions (slide 2), the classes of compounds that produce ions and the role of the number of ions produced and their charges (slide 3).
   c. Conductivity is the reciprocal of resistivity. Many have probably seen this demonstration of the property of conductivity (slide 4). Relate it to resistivity.
   d. Representation of a Wheatstone bridge which measures resistance, and a list of types of sensors (slide 5).
   e. Units used for resistance and conductance (slide 6).

f. Discuss electrode characteristics: one type of construction (slide 7), and the critical factors (slide 8). A "standard" cell is one in which electrodes are spaced exactly 1 cm apart and each has a surface area of 1 cm^2. Laboratory cells are usually not standard.

g. How to determine a cell constant for cells which are not standard (slide 9).

h. If a cell constant is calculated from standard cell conductance values, this formula converts meter results to the value that would have been obtained using a standard cell. The result is called "specific" conductance (slide 10).

i. Application of a cell constant has limits (slide 11).

j. Temperature is a critical factor affecting conductivity. (See figure 5 in outline). Therefore, results are to be reported as values that would be obtained at 25°C. This chart from 13th ed. Standard Methods (slide 12) demonstrates the significant effect of temperature by showing the factor required to convert results to a 25°C equivalent. Most meters give results which are temperature-compensated, so it is not necessary to convert the results.

k. Discuss the problems encountered in this measurement (slide 13).

l. Briefly note various applications of conductivity measurements. Details about these applications, including graphs as appropriate, are in the training manual outline (slides 14 and 15).

2. Laboratory - 50 minutes. Use the outline, "Calibration and Use of Conductivity Meter" or its equivalent.
   a. Assigment of training manual procedures

      1) Check instrument using II B and II C

      2) Obtain readings to calculate cell constants for the three probes using section III

         a) Note most accurate part of scale

         b) Note Cell Information and Table 1.

      3) Obtain readings for 3 samples. Note section IV and Table 2

      4) As time permits, calculate cell constants (note IB.1.) and apply to readings for samples to get results to report.

   b. Student performance of assignment

3. Classroom Discussion - 10 minutes

   a. Students finish calculations and post results.

   b. Discuss results.

   c. Discuss any items from laboratory session.

d. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Cell constant for three probes

Three specific conductance samples

To minimize the number of conductivity meters and probes required, the laboratory session could be conducted twice with half the class doing other procedures requiring about the same time, e.g., Turbidity and Second Weighing of Solids.

A. For each station (no more than two persons per station):

1. Seven 400 ml beakers
2. One 600 ml beaker (probe rinse)
3. One box tissues
4. One thermometer
5. One conductivity meter with Wheatstone Bridge (Ex. Beckman Solu Bridge)
6. One 10,000 ohm resistor
7. Three conductivity probes - VS2, VS02, VS20, or equivalent
8. One squeeze bottle distilled water
9. One marking pencil
10. One apron for each student
11. One pair safety glasses for each student

B. Shared:

1. None

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition "Standard Methods", page 74.

A. Minimum amounts per station (no more than two persons per station):

1. Four hundred ml each of four potassium chloride standards - 0.1N, 0.01N, 0.001N, 0.0001N
2. Four hundred ml municipal wastewater treatment plant effluent sample. A synthetic sample could be used instead. See next.
3. Four hundred ml sample "A", the same concentration as the 0.01N KCl standard (or can mix standards).

4. Four hundred ml sample "B", the same concentration as the 0.001N KCl standard (or can mix standards).

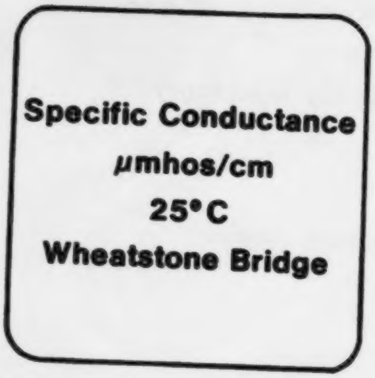
B. Prepare/obtain excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Fifteen slides, X-29: Specific Conductance - See next pages.

SERIES X-29: SPECIFIC CONDUCTANCE



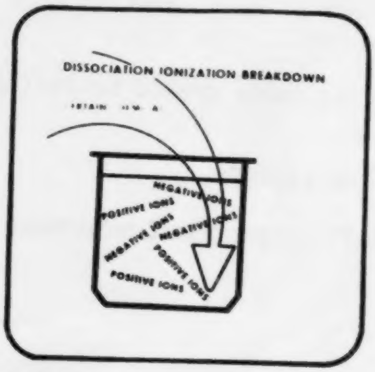
Specific Conductance

µmhos/cm

25°C

Wheatstone Bridge

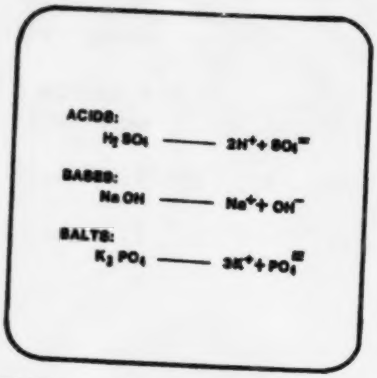
SLIDE 1



DISSOCIATION IONIZATION BREAKDOWN

NEGATIVE IONS
POSITIVE IONS
NEGATIVE IONS
NEGATIVE IONS
POSITIVE IONS
POSITIVE IONS

SLIDE 2



ACIDS:

H_2SO_4 —— $2H^+ + SO_4^=$

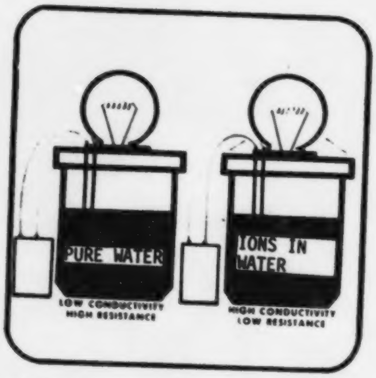
BASES:

$NaOH$ —— $Na^+ + OH^-$

SALTS:

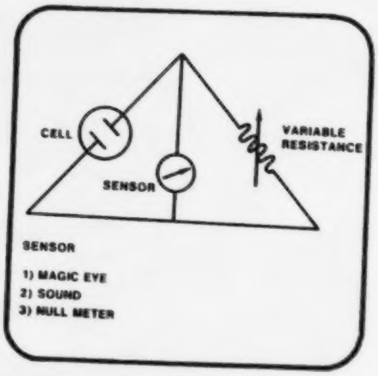
K_3PO_4 —— $3K^+ + PO_4^{\equiv}$

SLIDE 3



PURE WATER
LOW CONDUCTIVITY
HIGH RESISTANCE
IONS IN WATER
HIGH CONDUCTIVITY
LOW RESISTANCE

SLIDE 4

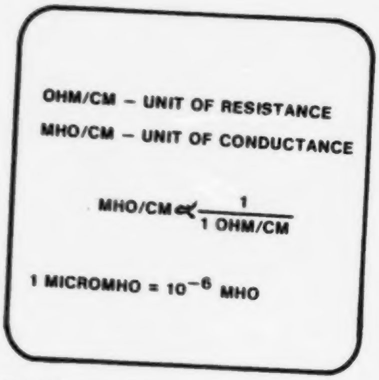


CELL
SENSOR
VARIABLE RESISTANCE

SENSOR

1) MAGIC EYE
2) SOUND
3) NULL METER

SLIDE 5



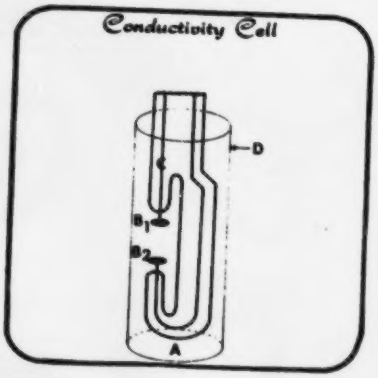
OHM/CM – UNIT OF RESISTANCE

MHO/CM – UNIT OF CONDUCTANCE

$$\text{MHO/CM} \propto \frac{1}{1\ \text{OHM/CM}}$$

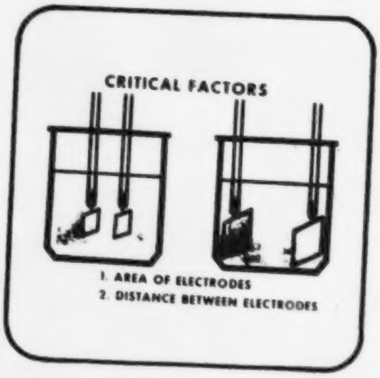
1 MICROMHO = 10^{-6} MHO

SLIDE 6



Conductivity Cell

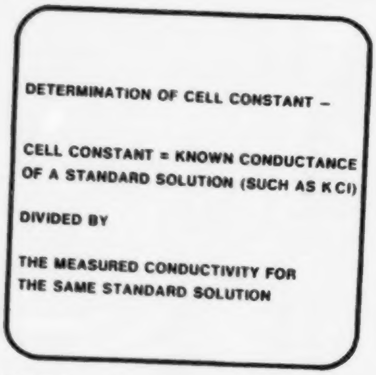
SLIDE 7



CRITICAL FACTORS

1. AREA OF ELECTRODES
2. DISTANCE BETWEEN ELECTRODES

SLIDE 8



DETERMINATION OF CELL CONSTANT –

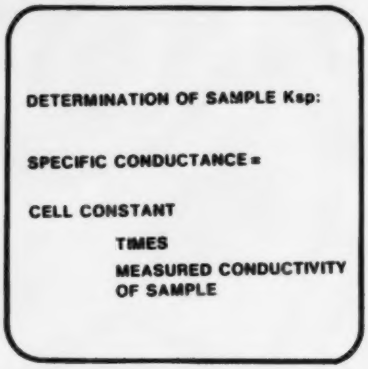
CELL CONSTANT = KNOWN CONDUCTANCE OF A STANDARD SOLUTION (SUCH AS KCl)

DIVIDED BY

THE MEASURED CONDUCTIVITY FOR THE SAME STANDARD SOLUTION

SLIDE 9

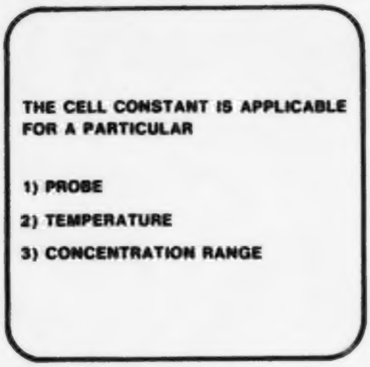
SERIES X-29: SPECIFIC CONDUCTANCE


DETERMINATION OF SAMPLE Ksp:

SPECIFIC CONDUCTANCE =

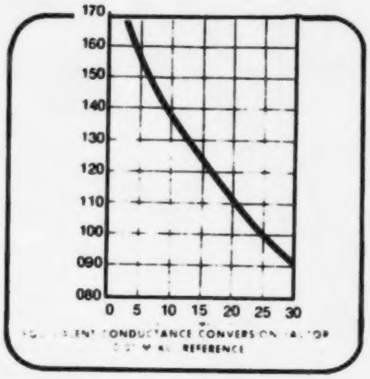
CELL CONSTANT
TIMES
MEASURED CONDUCTIVITY OF SAMPLE

SLIDE 10

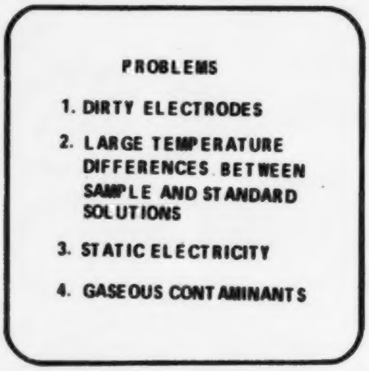

THE CELL CONSTANT IS APPLICABLE FOR A PARTICULAR

1) PROBE
2) TEMPERATURE
3) CONCENTRATION RANGE

SLIDE 11

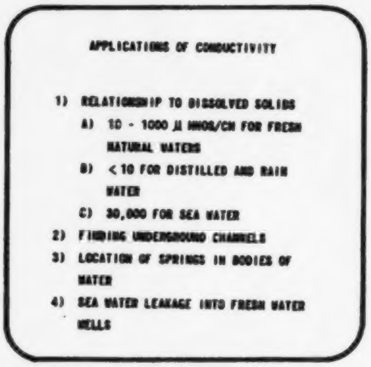


SLIDE 12


PROBLEMS

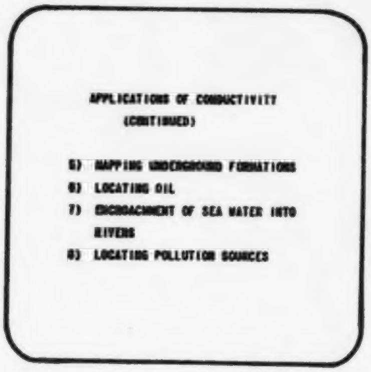
1. DIRTY ELECTRODES
2. LARGE TEMPERATURE DIFFERENCES BETWEEN SAMPLE AND STANDARD SOLUTIONS
3. STATIC ELECTRICITY
4. GASEOUS CONTAMINANTS

SLIDE 13


APPLICATIONS OF CONDUCTIVITY

1) RELATIONSHIP TO DISSOLVED SOLIDS
    A) 10 - 1000 μ MHOS/CM FOR FRESH NATURAL WATERS
    B) < 10 FOR DISTILLED AND RAIN WATER
    C) 30,000 FOR SEA WATER
2) FINDING UNDERGROUND CHANNELS
3) LOCATION OF SPRINGS IN BODIES OF WATER
4) SEA WATER LEAKAGE INTO FRESH WATER WELLS

SLIDE 14


APPLICATIONS OF CONDUCTIVITY (CONTINUED)

5) MAPPING UNDERGROUND FORMATIONS
6) LOCATING OIL
7) ENCROACHMENT OF SEA WATER INTO RIVERS
8) LOCATING POLLUTION SOURCES

SLIDE 15

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